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QUARTERLY REPORT

GTI PROJECT NUMBER 20916

Modeling of Microbial Induced Corrosion on Metallic Pipelines Resulting from Biomethane and the Integrity Impact of Biomethane on Non-Metallic Pipelines

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List Activities/Deliverables Completed During Reporting Period

	SCH Date	CMPL Date
Task #3: Lab Evaluation of Microbial Corrosion	03/31/2011	In Progress

- Completed testing of the charge mosaic membrane (CMM) for blocking bacteria migration from anode to cathode cell.
- Modified and assembled dual cell electrochemical test setup.
- The current test setup is ongoing using the dual cell to generate preliminary data.

Task 4#: Preliminary Microbial Corrosion Model Development 09/30/2012 In progress

- South West Research Institute (SWRI) signed an agreement with Ohio University (OU) to hire Prof. Tingyue Gu as a consultant for microbial corrosion model development.
- GTI, SWRI and OU reviewed the scope of work at OU to conduct vial tests at the anaerobic conditions that will support the model development.
- Testing anaerobic Bacillus licheniformis bacteria was conducted at OU and the preliminary test results were reported to SWRI and GTI.
- Testing on acid produced bacteria is ongoing.

Task #8: Perform Bounded Testing 9/30/2012 In Progress

- Completed collecting gas samples (raw landfill, processed landfill and raw dairy farm gases) from the field.
- Completed natural gas saturation test.
- Completed sample preparation and baseline testing for saturation test with processed landfill gas.
- Comparative testing (baseline without gas exposure) is ongoing.
- Comparative testing with natural gas exposure is ongoing.
- Saturation test with processed landfill gas is ongoing.

Technical Status

Task 3 - Lab Evaluation of Microbial Corrosion under Simulated Field Conditions

The focus of the work in this quarter was to solve the bacteria contamination in the cathode cell. It was indicated from the previous work that the migration of bacteria through the salt bridge connecting the anode and cathode cell was the major issue causing bacteria contamination in the cathode cell. Various types of filters were tested in the last quarter, however the results of those filters were not deemed sufficient for baceteria control. Bacteria in the anode cell were able to pass through the filter and populate the cathode cell in a short period of time so that the potential difference between the anode and cathode could not be maintained for to generate enough experimental data.

In this quarter, the ion exchange membranes were reviewed for being used in the salt bridge to block bacteria migration. A charge mosaic membrane (CMM) was chosen because it allows both anions and cations to migrate through the membrane, but blocks the bacteria migration. However, there is no commercially available product of this type of membrane. GTI used experimental CMM membrane (see Figure 1) that was developed by researchers in GTI's Basic Energy and Environmental Group.

The salt bridge was redesigned and assembled in order to install the CMM membrane and prevent airborne bacteria from contaminating each cell. A cam and groove hose coupling was used to install the CMM membrane, and the rubber gasket in the coupling created a tight seal to prevent liquid leakage. The membrane was then tested in the dual cell system (see Figure 2) for a month to validate its efficiency on blocking bacteria.

Both cells were filled with artificial growth medium (AGM) supplemented with 0.3% of Nutrient Broth (NB), and the anode cell was inoculated with a bacteria consortium (10%). The AGM recipe (minus nutrient broth) is shown in Table 1. The bacteria consortium was prepared by mixing the *B. licheniformis* culture and the enrichment culture of field condensate at the ratio of 1:10 (bacteria number). Two measurements of bacteria were taken during the test on each cell, and bacteria were found in the anode cell but not found in the cathode cell.

Modification was made on the dual cell electrochemical test setup to improve the sealing condition from the instrumentation ports and prevent airborne bacterial contamination, see Figure 3 and Figure 4 for the anode and cathode cell setup. Three positioning devices were designed and installed in the anode cell in order to position the micro-pH probe type very close to the anode surface without touching. The plastic fittings that were aged over the multiple autoclave cycles in the past were replaced with new fittings to ensure no leakage from the fittings.

The reference electrodes (Calomel reference electrode) and micro-pH probes were thoroughly cleaned and calibrated before testing. The anodes were polished to obtain a clean and fresh surface before they were assembled into the test cell. The test cells and all the parts were either autoclaved or treated by sterilizing and disinfecting solution (SSDS), and the cells and the instrumentations were assembled in the UV sterilization hood to prevent the contamination of the test setup.

The preliminary testing with the dual cell electrochemical test setup was started on Sept. 15, 2011, see Figure 5. 2L of sterile artificial growth medium supplemented with 0.3% of Nutrient Broth was pumped into anode and cathode cell respectively through a 0.2 micron membrane filter, and purged with filtered gas mixture (94.3% N_2-5%H_2-0.7%O_2) for 60 minutes. The anode cell was inoculated with bacterial consortium (10%), while cathode cell was kept abiotic. The temperatures of both cells are maintained at 30ºC with heat tape.

Monitoring the potentials and pH of the anodes and cathode were started after inoculation of bacteria using the ***nanoCorr™ Coupled Multielectrode Analyzer***. The experiment is ongoing, and a daily culture sample is taken to determine the concentration of planktonic bacteria using the plate count method with serial dilution in triplicate. When the concentration reaches 10^6 cells/ml, medium replacement in anode cell will be started. 10% of the culture volume in the anode cell is replaced daily with fresh growth medium filtered through a 0.2 micron membrane filter. The medium composition in anode cell is analyzed periodically. The headspace of anode and cathode cells is purged with gas mixture daily to maintain 0.7% oxygen condition.

Corrosion rate measurements will be taken after a steady potential difference (>10 mV) is generated between anode and cathode. The results will be reported in the next quarterly report.

Task 4 - Preliminary Microbial Corrosion Model Development

In this quarter, a consulting agreement between SwRI and Dr. Gu of Ohio University (OU) was formed. Later, GTI, SwRI and OU discussed and initiated OU to perform some short term experimental tests. The aim of the tests was to understand the mechanisms of microbial corrosion in the similar conditions that were defined by GTI for this study.

OU has produced a brief report to SwRI (and shared with GTI) based on the test results obtained so far. In these tests, MIC pitting was caused by the anaerobic respiration of Bacillus licheniformis (ATCC 14580) and nitrate was used as the terminal electron acceptor. Loose and fluffy biofilms on test coupons were observed. The initial culture medium pH was 7. After 7 days of anaerobic growth in the nitrate-containing medium, the pH dropped to 5.69 (50ºC) and 5.75 (37ºC) respectively. Pits were found and they were similar at both temperature. The largest pit depth was 45μm at 50ºC, and 35μm at 37ºC, see Figure 6 and Figure 7. They were much deeper than the 10μm pits typically found for SRB in one week tests. These pits suggest Bacillus licheniformis can be aggressive to pitting. This aspect will be taken into account for developing a microbial corrosion model for biogas gathering pipeline.

Tests with acid producing bacteria are on-going. Results will be reported in the next quarterly.

Task 8 - Perform Bounded Testing to Generate a Strong Example Data Set

The field trip to collect biogas samples was completed. Three gas samples were compressed to 1800psig including one raw landfill gas, one processed landfill gas and one raw dairy farm gas.

The first batch of saturation testing with natural gas was completed. The gas saturation progress was monitored by analyzing the gas components absorbed in the test samples (Headspace Test by Gas Chromatography (GC)). The saturation test was stopped after the gas components become stabilized in the test samples. Hardness and dimensional measurements were taken periodically after the test samples were removed from the pressure vessel. The material property tests were conducted after the hardness and dimensional measurements reached equilibrium.

A second saturation test with processed landfill gas was started on Sept. 8, 2011. The saturation test is ongoing, and the test duration is scheduled for three months. The baseline measurements of hardness and dimensional measurements were taken for the three test materials (SBR, NBR and MDPE) before they were put on test.

The samples for PENT (ASTM F1473 Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins) baseline comparative testing on the MDPE have been on test, and will be completed next quarter.

Biogas Samples

Three biogas samples (raw landfill, processed landfill and raw dairy farm gases) were collected from the field and delivered to GTI. Figure 8, Figure 9, and Figure 10 shows the gas collecting process for raw landfill, processed landfill and raw dairy farm gases respectively. The water vapor in the raw biogases was removed during the sample collection to prevent the gas chemistry change due to water condensing when the gases were compressed into the high pressure cylinders. Water will be added during the saturation test to recover the moisture level in the raw gases.

The chemical composition of the three gases collected from the field was analyzed by GC. The results are shown in Table 2. The hydrogen sulfide in the raw dairy gas sample was very low and it was not detected in the raw landfill gas sample. The lower hydrogen sulfide level in the raw gases might be due to the moisture removal process.

Natural Gas Saturation Test Results

Gas Saturation Progress (Headspace)

The gas saturation progress was monitoring by measuring the gas components in the test specimens by GC. The test results of total C4+, cycloalkanes and the total of Benzene, Toluene, Ethylbenzene, and Xylene (BTEX) during the natural gas saturation test (up to 1848 hours) are shown in Figure 11 through Figure 19. The test specimens were about saturated up to this time and the gas saturation test was terminated after 98 days (2352 hrs).

Outgas Progress

Hardness and dimensional data of three test specimens (#1, #2 and #3) were taken immediately after the samples were taken out of the pressure vessel after the natural gas saturation test was terminated. The measurements were taken on the specimens from each material (SBR, NBR, and MDPE) again at 18 hours. The final measurements of all the specimens were taken at 792 hours after gas saturation test.

Table 3 through Table 6 is the dimensional measurements on X and Y directions for baseline and 0, 18, and 792 hrs after gas saturation test. Table 7 through Table 10 list the dimensional measurements on Z direction for baseline and 0, 18, and 792 hours after gas saturation test.

The dimensional change at 0, 18, and 792 hrs after gas saturation test was calculated and plotted in Figure 20, Figure 21, and Figure 22 for SBR, NBR and MDPE respectively. It can be seen that the outgas process slowed down after 18 hours, and it was considered negligible for the measurements taken at 792 hours after the specimens were removed from natural gas saturation test.

Table 11 and Table 12 are the hardness measurements for SBR and NBR before natural gas saturation test and 0, 18 and 792 hours after the gas saturation test was completed. There was no major change of the hardness for both elastomeric materials before and after the gas saturation test, and the hardness change was not significant over the outgas progress.

The hardness of MDPE was measured before natural gas saturation test and 792 hours after the saturation test was completed. The hardness of MDPE after natural gas saturation was about the same as the baseline measurements.

Dimensional Change

The percentage of dimensional change of the test specimens were calculated using the final measurements at 792 hrs after the specimens were removed from natural gas saturation test:

where X_f, Y_f, and Z_f are the final measurements, and X_b, Y_b and Z_b are the baseline measurements of the test specimens at X, Y and Z directions.

Figure 23, Figure 24 and Figure 25 show the percentage of dimensional change of SBR, NBR and MDPE respectively after natural gas saturation test.

For SBR (see Figure 23), all the specimens shrank on the thickness (Z direction) and the average of the shrinkage of the specimens thickness is about 6%. Except for sample #2, one dimension on X and Y directions expanded and the other one shrank. The maximum dimensional expansion is less than 0.2% and the maximum shrinkage is less than 0.5%.

For NBR (see Figure 24), most of the test specimens shrank on both X and Y directions, except sample #3. The maximum shrinkage of the specimen dimension on X and Y directions is less than 0.4%. Sample #1, #2, and #3 showed significant shrinkage on Z direction, i.e., 3.3%, 8.4% and 6.3% for #1, #2 and #3 respectively. But the other three (#3, #4, and #5) have minor expansion (less than 0.3%) on Z direction. It is unknown yet what caused the variation of the thickness measurement on NBR test specimens.

For MDPE (see Figure 25), most of the specimens expanded on both X, Y and Z directions except #3 and #5. The maximum expansion are less than 0.9% on X and Y directions, and less than 0.2% on Z direction.

Hardness

Hardness testing was performed using a shore hardness testing apparatus. The test method being used is ASTM D2240. Five (5) measurements on three (3) replicates were recorded. Results for the natural gas exposure test are shown in Table 11.

Gas Saturation Test with Processed Landfill Gas

Test Specimens

Test specimens were prepared for the gas saturation test with processed landfill gas, see Table 14. The rubber samples were prepared by cutting specimens from the rubber sheet material to size. The plastic test specimens were machined to size from molded plaques. The tensile specimens were prepared by die cut (ASTM D638 Type IV). The compression test samples will be die cut from the 5.7"×2.25"×0.125" sheets after the gas saturation test. To perform hardness and compression test for SBR and NBR, the specimens with 0.125" thickness will be stacked to the required sample thickness (0.25" for hardness and 0.5" for compression) according to ASTM D2240 and ASTM D575. The dimensions for the PENT test (ASTM F1473 Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins) are 1.97"×0.98"×0.25".

The surface of the test specimens were cleaned with isopropyl alcohol, rinsed with water and dried in air after sample preparation. The specimens were marked by attaching an identifying pin.

Gas Saturation Test

Figure 26 shows the gas saturation test system. It consists of a main pressure vessel and a secondary vessel containing the head space samples. The test specimens were set on to the test cage and loaded into the pressure vessels. Figure 27 and Figure 28 show the apparatus for the specimens in the main and secondary vessels respectively.

The vessels were purged with the tested gas to get rid of the air and then a slow flow rate of 0.05 cc/min (0.18 cubic inches per hour) is being maintained during the saturation test. The main vessel will be purged again with the tested gas each week in order to refresh the gas.

The head space samples in the secondary vessel will be taken periodically to monitor the gas saturation progress. One sample of each test material (SBR, NBR and MDPE) will be taken at a time, and the samples will then be analyzed by gas chromatography (GC) to determine the gas components that have been absorbed into the test materials.

Dimensional Measurements

The baseline dimensional measurements were taken on the specimens before saturation test. The specimen surface was slightly scribed to draw lines along X and Y direction, see Figure 29 and the positions (x, y) of P_1, P_2 and P_3 were measured using optical microscope. The measurements were taken three times on each specimen and the specimen was repositioned before taking the measurements to obtain the repeatability of the measurements. The distances of P_1P_2 and P_2P_3 were calculated as the baseline dimensional measurements for X and Y directions respectively, see Table 15. Three data points were taken at the center of each specimen to obtain the average thickness measurements (dimension in Z direction), see Table 16.

The dimensional measurements will be repeated on the test specimens after the saturation test to calculate the dimensional change before and after gas saturation.

Hardness

Hardness measurements were taken using a shore hardness testing apparatus. The test method being used is ASTM D2240. An average of five (5) measurements on three (3) replicates for SBR and NBR, and MDPE were recorded. Results to date are shown in Table 17.

Table 1. Artificial Growth Medium Recipe for Corrosion Experiments

Macronutrients	**Milligram per L**
NH_4HCO_3	400 mg
$Na_2HPO_4 \cdot H_2O$	30 mg
K_2SO_4	20 mg
$CaCl_2$	9 mg
$FeCl_2 \cdot 4H_2O$	5 mg
$MgSO_4 \cdot 7H_2O$	3 mg
100X Trace Elements stock (add 10 ml to 1 L)	**Milligram per 100 mL**
$MnCl_2 \cdot 4H_2O$	180
$CoCl_2 \cdot 6H_2O$	270
H_3BO_3	50
$CuCl_2 \cdot 2H_2O$	24
$NaMoO_4 \cdot 2H_2O$	23
$ZnCl_2$	19
100X Vitamins stock (add 10 ml to 1 L) *from ATCC Vitamin Supplement Formulation Catalog No: MD-VS	**Milligram per 100 mL**
Biotin	0.2
Folic Acid	0.2
Pyridoxine Hydrochloride	1.0
Riboflavin	0.5
Thiamin	0.5
Nicotinic Acid	0.5
B_{12}	0.01
p-Aminobenzoic Acid	0.5
Thioctic Acid	0.5
Calcium pantothenate	0.5
Monopotassium phosphate	0.5

Table 2. The Chemistry of the Gas Samples Collected from the Field

Group #	Concentration	Gas Constituents	Raw Landfill Gas	Processed Landfill gas	Raw Dairy Gas
1	(mol%)	Hydrogen	0.81	1.65	ND
	(mol%)	Nitrogen	3.19	3.76	1.44
2	(mol%)	Carbon dioxide	40.5	2.46	40.0
	(mol%)	Oxygen	0.59	0.42	0.12
	(mol%)	Hydrogen sulfide	ND	ND	0.193
	(ppmv)	Sulfur dioxide	ND	ND	0.62
3	(mol%)	Methane	54.9	91.7	58.2
	(mol%)	Propane	0.003	ND	ND
	(mol%)	n-Hexane	0.0025	BDL	0.0001
	(mol%)	n-Heptane	0.0017	BDL	BDL
	(mol%)	n-Octane	0.0017	BDL	0.0001
	(mol%)	Nonane	0.0022	BDL	0.002
	(mol%)	n-Decane	0.0025	BDL	0.0005
	(mol%)	Undecane	0.0005	BDL	0.0001
4	(mol%)	Cyclopentane	0.0001	BDL	BDL
	(mol%)	Methylcyclopentane	0.0001	BDL	BDL
	(mol%)	Cyclohexane	0.0002	BDL	BDL
	(mol%)	Methylcycohexane	0.0002	BDL	BDL
5	(mol%)	Ethene	0.002	ND	ND
	(mol%)	Propene	0.003	ND	ND
6	(mol%)	Benzene	0.0016	BDL	0.0002
	(mol%)	Toluene	0.0020	BDL	0.0006
	(mol%)	Ethylbenzene	0.0003	BDL	0.0001
	(mol%)	m,p-Xylene	0.0004	BDL	0.0001
	(mol%)	o-Xylene	0.0001	BDL	BDL
	(mol%)	Styrene	0.0001	BDL	BDL
	(mol%)	C3 benzene	0.0004	BDL	0.0001

Note:
BDL: Below Detection Limit
ND: Not Detected

Table 2. The Chemistry of the Gas Samples Collected from the Field (Continued)

Group #	Gas Constituents (ppmv)	Raw Landfill Gas	Processed Landfill Gas	Raw Dairy Gas
7	Carbonyl Sulfide	0.13	ND	2.79
	Carbon Disulfide	0.19	ND	0.30
	Methyl Mercaptan	ND	ND	2.31
	Ethyl Mercaptan	ND	ND	0.14
	t-Butyl Mercaptan	ND	ND	0.25
	Dimethyl Sulfide	18.7	0.24	1.29
	Methyl Ethyl Sulfide	0.19	ND	ND
	Dimethyl Disulfide	1.29	ND	0.06
	Methyl Ethyl Disulfide	0.04	ND	ND
	Dimethyl Trisulfide	0.08	ND	ND
	Thiophene	0.48	ND	ND
	C1-Thiophenes	0.32	ND	0.07
8	Dichlorodifluoromethane	1.16	BDL	BDL
	1,2-Dichlorotetrafluoroethane	0.15	BDL	BDL
	Chloromethane	0.20	BDL	BDL
	Dichloromethane	0.33	BDL	BDL
	Vinylchloride	0.38	BDL	BDL
	Chloroethane	0.40	BDL	BDL
	1,2-Dichloroethene	0.45	BDL	BDL
	Trichloroethene	0.32	BDL	BDL
	Tetrachloroethene	0.55	BDL	BDL
9	1,1,3,3,-Tetramethyldisiloxane	<0.10	<0.10	<0.10
	Pentamethyldisiloxane	<0.10	<0.10	<0.10
	Hexamethyldisilane	<0.10	<0.10	<0.10
	Hexamethyldisiloxane (L_2, MM)	0.30	<0.10	<0.10
	Octamethyltrisiloxane (L3, MDM)	<0.07	<0.07	<0.07
	Octamethylcyclotetrasiloxane (D4)	<0.05	<0.05	<0.05
	Decamthyltetrasiloxane (L_4, MD_2M)	<0.05	<0.05	<0.05
	Decamethylcyclopentasiloxane (D5)	<0.04	<0.04	<0.04
	Dodecamthylpentasiloxane (L_5, MD_3M)	<0.04	<0.04	<0.04
	Dodecamethylcyclohexasiloxane	<0.03	<0.03	<0.03

Note:
BDL: Below Detection Limit
ND: Not Detected

Table 3. Dimensional Measurements (X and Y Direction) Before Natural Gas Saturation Test (Baseline)

Materials	Specimen #	Measurements (mm)									
		X (P_1P_2)					Y (P_2P_3)				
		1	2	3	AVG	STDEV	1	2	3	AVG	STDEV
SBR	1	13.317	13.332	13.340	13.329	0.012	12.962	12.948	12.964	12.958	0.009
	2	13.097	13.098	13.104	13.099	0.004	13.464	13.473	13.483	13.474	0.009
	3	13.381	13.384	13.371	13.379	0.007	13.162	13.156	13.147	13.155	0.008
	4	12.959	12.941	12.945	12.948	0.009	13.642	13.634	13.636	13.637	0.005
	5	13.277	13.292	13.273	13.281	0.010	13.387	13.386	13.366	13.380	0.012
	6	13.261	13.249	13.256	13.255	0.006	13.037	13.033	13.035	13.035	0.002
NBR	1	12.462	12.457	12.462	12.460	0.003	13.211	13.225	13.231	13.223	0.010
	2	13.034	13.044	13.043	13.040	0.005	13.083	13.103	13.110	13.099	0.014
	3	13.071	13.073	13.067	13.070	0.003	13.361	13.377	13.392	13.377	0.016
	4	12.968	12.979	12.986	12.978	0.009	13.370	13.375	13.380	13.375	0.005
	5	13.488	13.480	13.489	13.486	0.005	13.130	13.140	13.154	13.141	0.012
	6	13.188	13.202	13.208	13.199	0.010	13.023	13.040	13.039	13.034	0.010
MDPE	1	12.855	12.859	12.853	12.855	0.003	12.614	12.608	12.607	12.610	0.004
	2	12.645	12.633	12.654	12.644	0.011	12.821	12.796	12.826	12.814	0.016
	3	12.469	12.471	12.466	12.469	0.003	12.829	12.833	12.838	12.834	0.004
	4	12.568	12.576	12.566	12.570	0.005	12.795	12.790	12.774	12.787	0.011
	5	12.497	12.493	12.501	12.497	0.004	12.379	12.355	12.347	12.360	0.017
	6	12.657	12.667	12.659	12.661	0.005	12.344	12.339	12.346	12.343	0.004

Table 4. Dimensional Measurements (X and Y Direction) After Specimens Taken out of Natural Gas Saturation Chamber (0 hr)

Materials	Specimen #	Measurements (mm)									
		X (P1P2)					Y (P2P3)				
		1	2	3	Ave	STDEV	1	2	3	Ave	STDEV
SBR	1	13.341	13.340	13.342	13.341	0.001	12.922	12.918	12.925	12.922	0.004
	2	13.053	13.059	13.056	13.056	0.003	13.461	13.465	13.466	13.464	0.003
	3	13.386	13.392	13.391	13.390	0.003	13.111	13.118	13.111	13.113	0.004
NBR	1	12.466	12.458	12.455	12.459	0.006	13.236	13.239	13.234	13.237	0.002
	2	12.994	13.005	13.015	13.005	0.011	13.094	13.109	13.115	13.106	0.011
	3	13.048	13.049	13.056	13.051	0.004	13.389	13.401	13.402	13.397	0.007
MDPE	1	12.889	12.896	12.881	12.889	0.007	12.649	12.642	12.633	12.641	0.008
	2	12.666	12.651	12.687	12.668	0.018	12.884	12.861	12.864	12.870	0.013
	3	12.487	12.490	12.497	12.491	0.005	12.863	12.888	12.905	12.885	0.021

Table 5. Dimensional Measurements (X and Y Direction) After Specimens Taken out of Natural Gas Saturation Chamber (18 hrs)

Materials	Specimen #	Measurements (mm)									
		X (P1P2)					Y (P2P3)				
		1	2	3	Ave	STDEV	1	2	3	Ave	STDEV
SBR	1	13.344	13.337	13.336	13.339	0.004	12.920	12.913	12.910	12.914	0.005
	2	13.046	13.057	13.053	13.052	0.006	13.457	13.466	13.462	13.462	0.005
	3	13.370	13.386	13.384	13.380	0.009	13.103	13.100	13.113	13.105	0.007
NBR	1	12.447	12.456	12.459	12.454	0.006	13.227	13.226	13.228	13.227	0.001
	2	12.995	13.004	13.008	13.002	0.007	13.097	13.112	13.110	13.107	0.008
	3	13.029	13.047	13.045	13.040	0.010	13.384	13.392	13.394	13.390	0.006
MDPE	1	12.899	12.896	12.872	12.889	0.015	12.632	12.634	12.345	12.537	0.166
	2	12.675	12.681	12.663	12.673	0.009	12.870	12.876	12.864	12.870	0.006
	3	12.481	12.472	12.464	12.473	0.009	12.870	12.869	12.861	12.867	0.005

Table 6. Dimensional Measurements (X and Y Direction) After Specimens Taken out of Natural Gas Saturation Chamber (792 hrs)

Materials	Specimen #	Measurements (mm)									
		X (P1P2)					Y (P2P3)				
		1	2	3	Ave	STDEV	1	2	3	Ave	STDEV
SBR	1	13.335	13.331	13.329	13.332	0.003	12.900	12.902	12.916	12.906	0.009
	2	13.051	13.052	13.052	13.052	0.001	13.460	13.464	13.462	13.462	0.002
	3	13.377	13.386	13.390	13.384	0.007	13.099	13.098	13.105	13.101	0.004
	4	12.930	12.926	12.943	12.933	0.009	13.638	13.661	13.660	13.653	0.013
	5	13.300	13.306	13.305	13.304	0.003	13.305	13.321	13.315	13.313	0.008
	6	13.236	13.233	13.235	13.234	0.002	13.037	13.046	13.047	13.044	0.006
NBR	1	12.453	12.446	12.453	12.451	0.004	13.201	13.217	13.213	13.210	0.008
	2	12.991	13.002	13.008	13.000	0.009	13.091	13.096	13.101	13.096	0.005
	3	13.029	13.037	13.043	13.036	0.007	13.385	13.394	13.384	13.387	0.006
	4	12.970	12.970	12.986	12.975	0.009	13.348	13.353	13.344	13.348	0.005
	5	13.469	13.479	13.481	13.476	0.006	13.118	13.135	13.130	13.127	0.009
	6	13.146	13.158	13.153	13.152	0.006	13.027	13.033	13.041	13.033	0.007
MDPE	1	12.875	12.876	12.880	12.877	0.003	12.633	12.616	12.622	12.624	0.009
	2	12.648	12.635	12.651	12.644	0.009	12.842	12.841	12.837	12.840	0.003
	3	12.459	12.474	12.455	12.463	0.010	12.868	13.098	12.861	12.942	0.135
	4	12.567	12.577	12.573	12.573	0.005	12.796	12.811	12.797	12.802	0.009
	5	12.481	12.458	12.456	12.465	0.014	12.374	12.343	12.343	12.353	0.018
	6	12.659	12.658	12.664	12.660	0.003	12.366	12.376	12.361	12.367	0.008

Table 7. Dimensional Measurements (Z Direction) Before Natural Gas Saturation Test (Baseline)

Materials	Specimen #	Measurements (mm)						
		1	2	3	4	5	AVG	STDEV
MDPE	1	6.398	6.388	6.385	6.392	6.388	6.390	0.005
	2	6.399	6.39	6.396	6.4	6.392	6.395	0.004
	3	6.403	6.407	6.396	6.393	6.394	6.399	0.006
	4	6.400	6.388	6.392	6.388	6.381	6.390	0.007
	5	6.399	6.398	6.407	6.408	6.401	6.403	0.005
	6	6.400	6.383	6.384	6.395	6.388	6.390	0.007
NBR	1	2.986	3.006	2.994	2.999	2.986	2.994	0.009
	2	3.437	3.428	3.429	3.476	3.444	3.443	0.020
	3	3.102	3.112	3.109	3.104	3.104	3.106	0.004
	4	3.082	3.093	3.09	3.097	3.092	3.091	0.006
	5	3.092	3.095	3.109	3.093	3.093	3.096	0.007
	6	3.435	3.435	3.462	3.437	3.471	3.448	0.017
SBR	1	3.370	3.36	3.373	3.375	3.375	3.371	0.006
	2	3.352	3.356	3.376	3.353	3.364	3.360	0.010
	3	3.369	3.368	3.371	3.373	3.36	3.368	0.005
	4	3.386	3.381	3.381	3.372	3.379	3.380	0.005
	5	3.358	3.347	3.365	3.365	3.365	3.360	0.008
	6	3.389	3.391	3.373	3.396	3.39	3.388	0.009

Table 8. Dimensional Measurements (Z Direction) After Specimens Taken out of Natural Gas Saturation Chamber (0 hrs)

Materials	Specimen #	Measurements (mm)				
		1	2	3	AVG	STDEV
MDPE	1	6.399	6.4	6.401	6.400	0.001
	2	6.401	6.399	6.395	6.398	0.003
	3	6.383	6.4	6.394	6.392	0.009
NBR	1	2.798	2.798	2.799	2.798	0.001
	2	3.143	3.203	3.204	3.183	0.035
	3	2.978	2.972	2.977	2.976	0.003
SBR	1	3.118	3.204	3.160	3.161	0.043
	2	3.151	3.266	3.216	3.211	0.058
	3	3.146	3.278	3.168	3.197	0.071

Table 9. Dimensional Measurements (Z Direction) After Specimens Taken out of Natural Gas Saturation Chamber (18 hrs)

Materials	Specimen #	Measurements (mm)				
		1	2	3	AVG	STDEV
MDPE	1	6.402	6.400	6.401	6.401	0.001
	2	6.399	6.399	6.398	6.399	0.001
	3	6.394	6.395	6.395	6.395	0.001
NBR	1	2.842	2.969	2.877	2.896	0.066
	2	3.146	3.125	3.187	3.153	0.032
	3	2.943	2.842	2.945	2.910	0.059
SBR	1	3.099	3.133	3.195	3.142	0.049
	2	3.144	3.136	3.193	3.158	0.031
	3	3.148	3.183	3.149	3.160	0.020

Table 10. Dimensional Measurements (Z Direction) After Specimens Taken out of Natural Gas Saturation Chamber (792 hrs)

Materials	Specimen #	Measurements (mm)				
		1	2	3	AVG	STDEV
MDPE	1	6.401	6.4	6.401	6.401	0.001
	2	6.4	6.399	6.398	6.399	0.001
	3	6.396	6.395	6.395	6.395	0.001
	4	6.399	6.389	6.391	6.393	0.005
	5	6.398	6.398	6.407	6.401	0.005
	6	6.4	6.399	6.387	6.395	0.007
NBR	1	2.842	2.969	2.877	2.896	0.066
	2	3.146	3.125	3.187	3.153	0.032
	3	2.943	2.842	2.945	2.910	0.059
	4	3.09	3.097	3.092	3.093	0.004
	5	3.109	3.093	3.093	3.098	0.009
	6	3.462	3.437	3.471	3.457	0.018
SBR	1	3.099	3.133	3.195	3.142	0.049
	2	3.144	3.136	3.193	3.158	0.031
	3	3.148	3.183	3.149	3.160	0.020
	4	3.118	3.204	3.16	3.161	0.043
	5	3.151	3.266	3.216	3.211	0.058
	6	3.146	3.278	3.168	3.197	0.071

Table 11. Hardness (Shore A) Data for SBR before and After Natural Gas Saturation Test

Measurement		Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6
Baseline	1	71	70	71	71	71	71
	2	69	71	71	71	71	71
	3	71	69	70	70	71	71
	4	70	70	71	70	70	71
	5	70	70	70	70	70	70
	Average	70.2	70	70.6	70.4	70.6	70.8
	Stdev	0.837	0.707	0.548	0.548	0.548	0.447
0 hrs	1	71	70	71	71	71	71
	2	69	71	72	72	70	70
	3	71	71	70	70	72	71
	4	71	70	71	70	70	71
	5	70	70	70	70	72	71
	Average	70.4	70.4	70.8	70.6	71	70.8
	Stdev	0.894	0.548	0.837	0.894	1.000	0.447
18 hrs	1	70	71	70	70	71	71
	2	70	71	70	71	70	70
	3	71	70	70	71	71	71
	4	71	71	70	71	70	70
	5	70	70	71	71	71	71
	Average	70.4	70.6	70.2	70.8	70.6	70.6
	Stdev	0.548	0.548	0.447	0.447	0.548	0.548
792 hrs	1	71	70	71	71	71	71
	2	69	71	72	72	70	70
	3	71	71	70	70	72	71
	4	71	70	71	70	70	71
	5	70	70	70	70	72	71
	Average	70.4	70.4	70.8	70.6	71	70.8
	Stdev	0.894	0.548	0.837	0.894	1.000	0.447

Table 12. Hardness (Shore A) Data for NBR before and After Natural Gas Saturation Test

Measurement		Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6
Baseline	1	70	70	70	70	71	71
	2	70	70	70	70	71	71
	3	70	70	70	70	71	71
	4	70	70	70	70	71	71
	5	70	70	70	70	71	71
	Average	70	70	70	70	71	71
	Stdev	0	0	0	0	0	0
0 hrs	1	70	70	70	70	71	71
	2	71	70	70	70	71	71
	3	70	70	70	70	71	71
	4	70	70	70	70	71	71
	5	71	70	70	70	71	72
	Average	70.4	70	70	70	71	71.2
	Stdev	0.548	0.000	0.000	0.000	0.000	0.447
18 hrs	1	70	70	70	70	71	71
	2	70	70	70	70	71	71
	3	70	70	70	70	70	71
	4	70	70	70	70	71	71
	5	70	70	70	70	71	71
	Average	70	70	70	70	70.8	71
	Stdev	0.000	0.000	0.000	0.000	0.447	0.000
792 hrs	1	70	70	70	70	71	71
	2	71	70	70	70	71	71
	3	70	70	70	70	71	71
	4	70	70	70	70	71	71
	5	71	70	70	70	71	72
	Average	70.4	70	70	70	71	71.2
	Stdev	0.548	0.000	0.000	0.000	0.000	0.447

Table 13. Hardness (Shore D) Data for MDPE before and After Natural Gas Saturation Test

Measurement		Sample #1	Sample #2
Baseline	1	34	33
	2	32	33
	3	32	34
	4	34	33
	5	33	32
	Average	33	33
	Stdev	1.000	0.707
792 hrs	1	33	33
	2	33	33
	3	32	33
	4	33	34
	5	33	32
	Average	32.8	33
	Stdev	0.447	0.707

Table 14. Summary of the Test Specimens for the Gas Saturation Test with Processed Landfill Gas

Length×Width (inch)	Thickness (inch)	Test	NBR	SBR	PE2708
1×1	0.25	Dimensional Change	NA	NA	6
	0.125		6	6	NA
5.75×2.25	0.25	Hardness	NA	NA	2
	0.125	Hardness & Compression	8	8	NA
2×1.625	0.125	Hardness (Out Gas)	6	6	NA
4.5×0.75	0.125	Tensile	8	8	NA
	0.16		NA	NA	7
1.97×0.98	0.25	Slow Crack Growth	NA	NA	6
0.25×0.25	0.25	Head Space	NA	NA	6
0.5×0.5	0.125		6	6	NA

Table 15. Dimensional Measurements (X and Y Direction) Before Processed Landfill Gas Saturation Test (Baseline)

Materials	Specimen #	Measurements (mm)									
		X (P1P2)					Y (P2P3)				
		1	2	3	Ave	STDEV	1	2	3	Ave	STDEV
SBR	1	12.454	12.440	12.442	12.445	0.008	12.271	12.264	12.260	12.265	0.006
	2	12.233	12.236	12.238	12.236	0.003	12.975	12.973	12.971	12.973	0.002
	3	12.666	12.673	12.674	12.671	0.004	12.225	12.223	12.222	12.223	0.002
	4	12.388	12.386	12.394	12.390	0.004	12.776	12.597	12.600	12.658	0.102
	5	12.272	12.282	12.279	12.278	0.005	12.208	12.205	12.211	12.208	0.003
	6	12.497	12.497	12.493	12.496	0.002	12.477	13.470	12.478	12.808	0.573
NBR	1	12.305	12.320	12.321	12.315	0.009	12.679	12.686	12.699	12.688	0.010
	2	12.333	12.337	12.325	12.332	0.006	12.233	12.240	12.210	12.228	0.016
	3	11.914	11.922	11.918	11.918	0.004	11.523	11.528	11.536	11.529	0.007
	4	12.600	12.604	12.595	12.599	0.005	12.568	12.564	12.563	12.565	0.003
	5	12.522	12.520	12.525	12.522	0.003	12.664	12.676	12.673	12.671	0.006
	6	12.121	12.121	12.100	12.114	0.012	12.024	12.030	12.016	12.023	0.007
MDPE	1	12.479	12.489	12.488	12.485	0.006	12.047	12.044	12.057	12.049	0.007
	2	12.800	12.807	13.035	12.881	0.134	12.275	12.275	12.250	12.267	0.015
	3	12.401	12.409	12.409	12.407	0.005	13.532	12.946	12.944	13.141	0.339
	4	11.858	11.849	11.853	11.853	0.005	12.008	12.020	12.015	12.014	0.006
	5	11.864	11.862	11.856	11.861	0.004	11.725	11.725	11.742	11.731	0.010
	6	11.699	11.691	11.688	11.693	0.006	11.241	11.240	11.241	11.241	0.001

Table 16. Dimensional Measurements (Z Direction) Before Processed Landfill Gas Saturation Test (Baseline)

Materials	Specimen #	Measurements (inch)				
		1	2	3	AVG	STDEV
MDPE	1	0.2475	0.2476	0.2475	0.248	0.000
	2	0.249	0.2491	0.249	0.249	0.000
	3	0.2484	0.2485	0.2484	0.248	0.000
	4	0.2483	0.248	0.2482	0.248	0.000
	5	0.2483	0.248	0.248	0.248	0.000
	6	0.2486	0.2485	0.2485	0.249	0.000
NBR	1	0.1132	0.1139	0.1139	0.114	0.000
	2	0.1127	0.113	0.113	0.113	0.000
	3	0.1126	0.1136	0.1136	0.113	0.001
	4	0.1133	0.1131	0.1131	0.113	0.000
	5	0.1134	0.1136	0.1136	0.114	0.000
	6	0.1136	0.1132	0.1132	0.113	0.000
SBR	1	0.1131	0.111	0.1148	0.113	0.002
	2	0.1151	0.1132	0.1148	0.114	0.001
	3	0.113	0.1131	0.1131	0.113	0.000
	4	0.1125	0.1129	0.1132	0.113	0.000
	5	0.1136	0.1137	0.1135	0.114	0.000
	6	0.1135	0.1133	0.1138	0.114	0.000

Table 17. Hardness Data for SBR, NBR and MDPE before Processed Landfill Gas Saturation Test (Baseline)

Measurement		Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Sample #7	Sample #8
SBR	1	69	69	69	69	71	70	69	69
	2	70	70	69	70	69	70	70	70
	3	69	70	69	69	70	70	70	70
	4	70	70	69	69	70	69	69	69
	5	70	69	70	70	70	70	70	70
	Average	69.6	69.6	69.2	69.4	70	69.8	69.6	69.6
	Stdev	0.548	0.548	0.447	0.548	0.707	0.447	0.548	0.548
NBR	1	70	70	70	70	69	70	69	71
	2	70	70	70	69	70	70	69	70
	3	70	70	70	69	70	70	69	69
	4	70	70	70	69	70	70	69	70
	5	70	70	70	70	70	70	69	70
	Average	70	70	70	69.4	69.8	70	69	70
	Stdev	0.000	0.000	0.000	0.548	0.447	0.000	0.000	0.707
MDPE	1	35	33	NA	NA	NA	NA	NA	NA
	2	33	35	NA	NA	NA	NA	NA	NA
	3	34	33	NA	NA	NA	NA	NA	NA
	4	35	35	NA	NA	NA	NA	NA	NA
	5	34	33	NA	NA	NA	NA	NA	NA
	Average	34.2	33.8	NA	NA	NA	NA	NA	NA
	Stdev	0.837	1.095	NA	NA	NA	NA	NA	NA

Figure 1. Charge Mosaic Membrane (CMM)

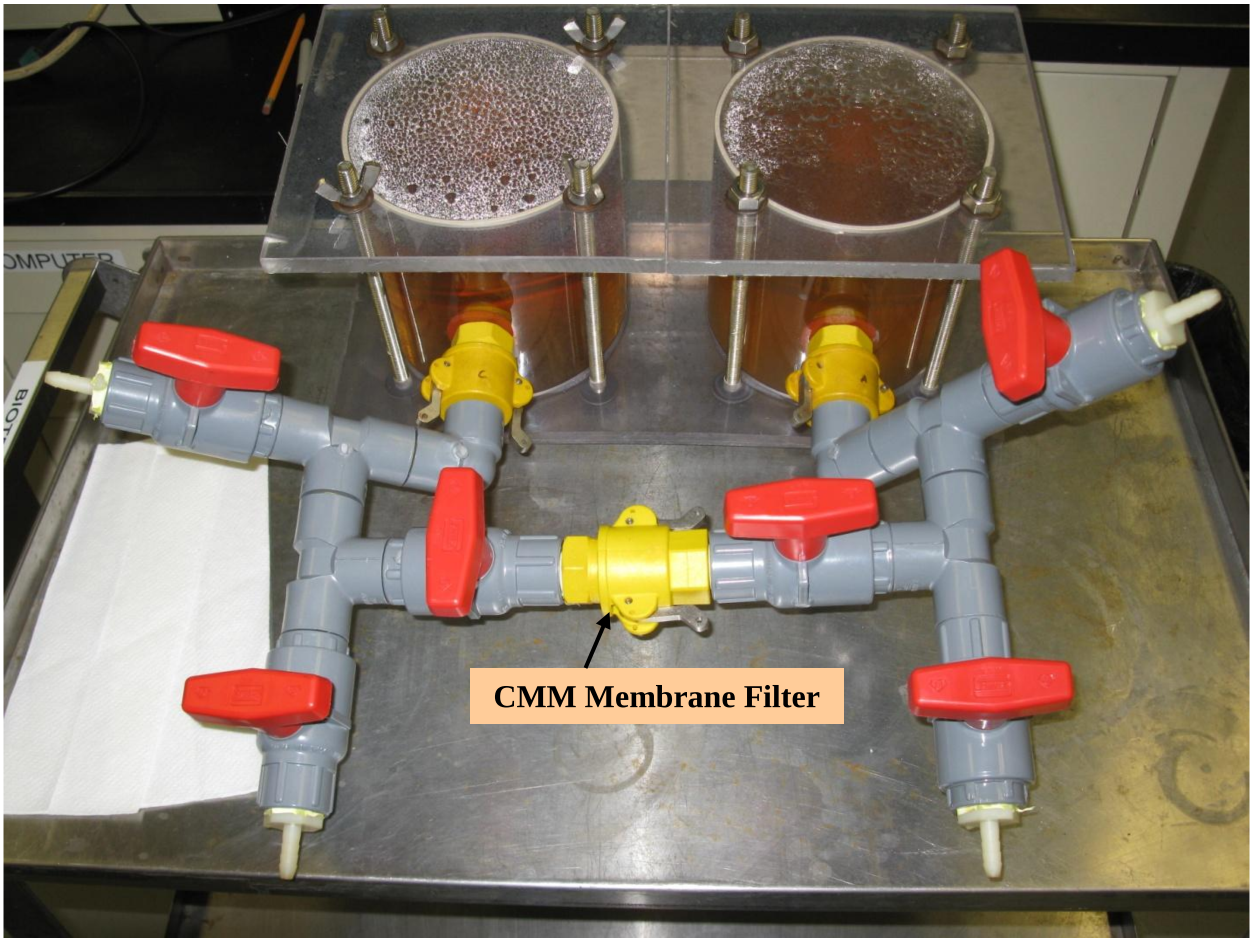
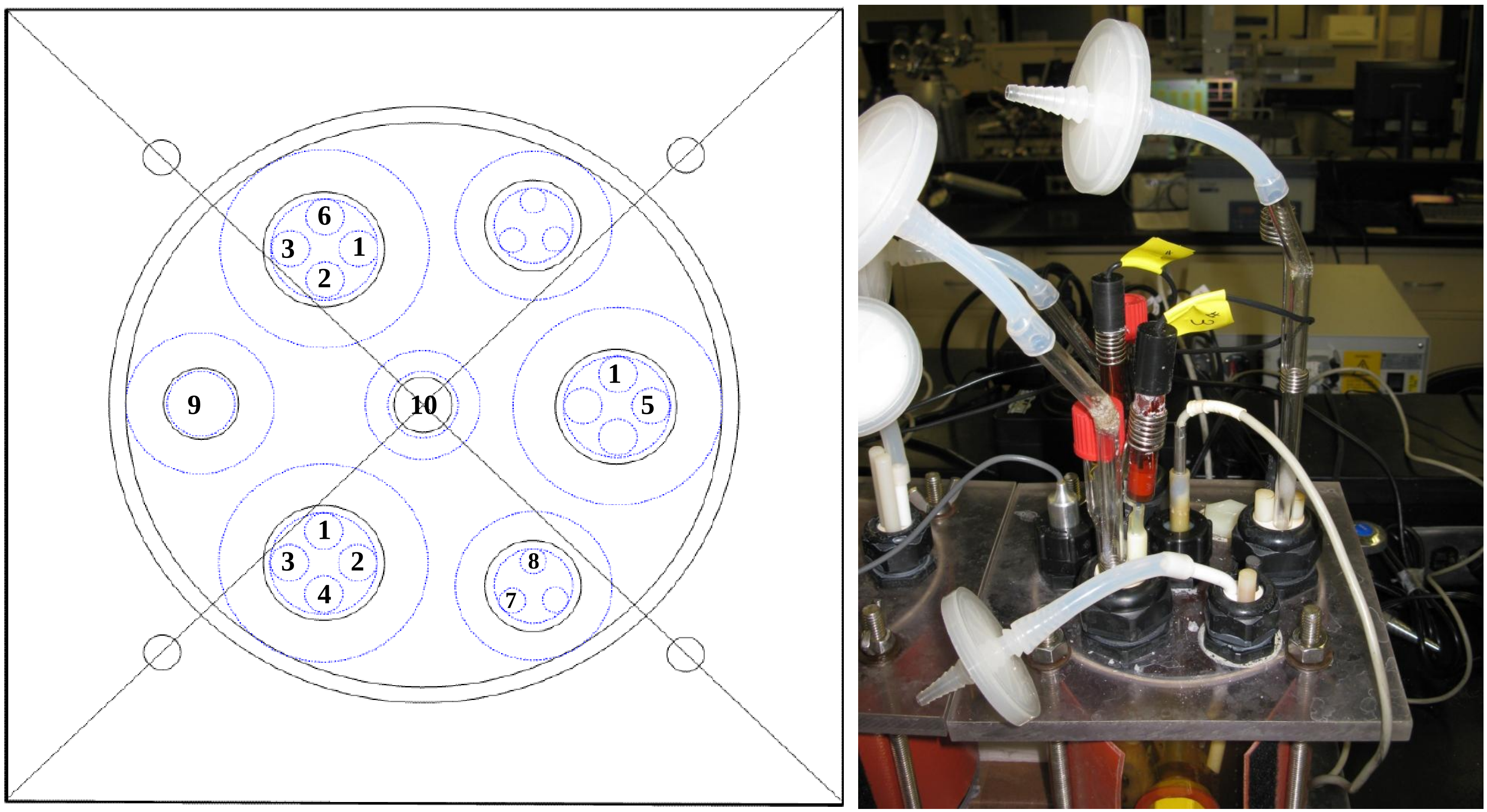
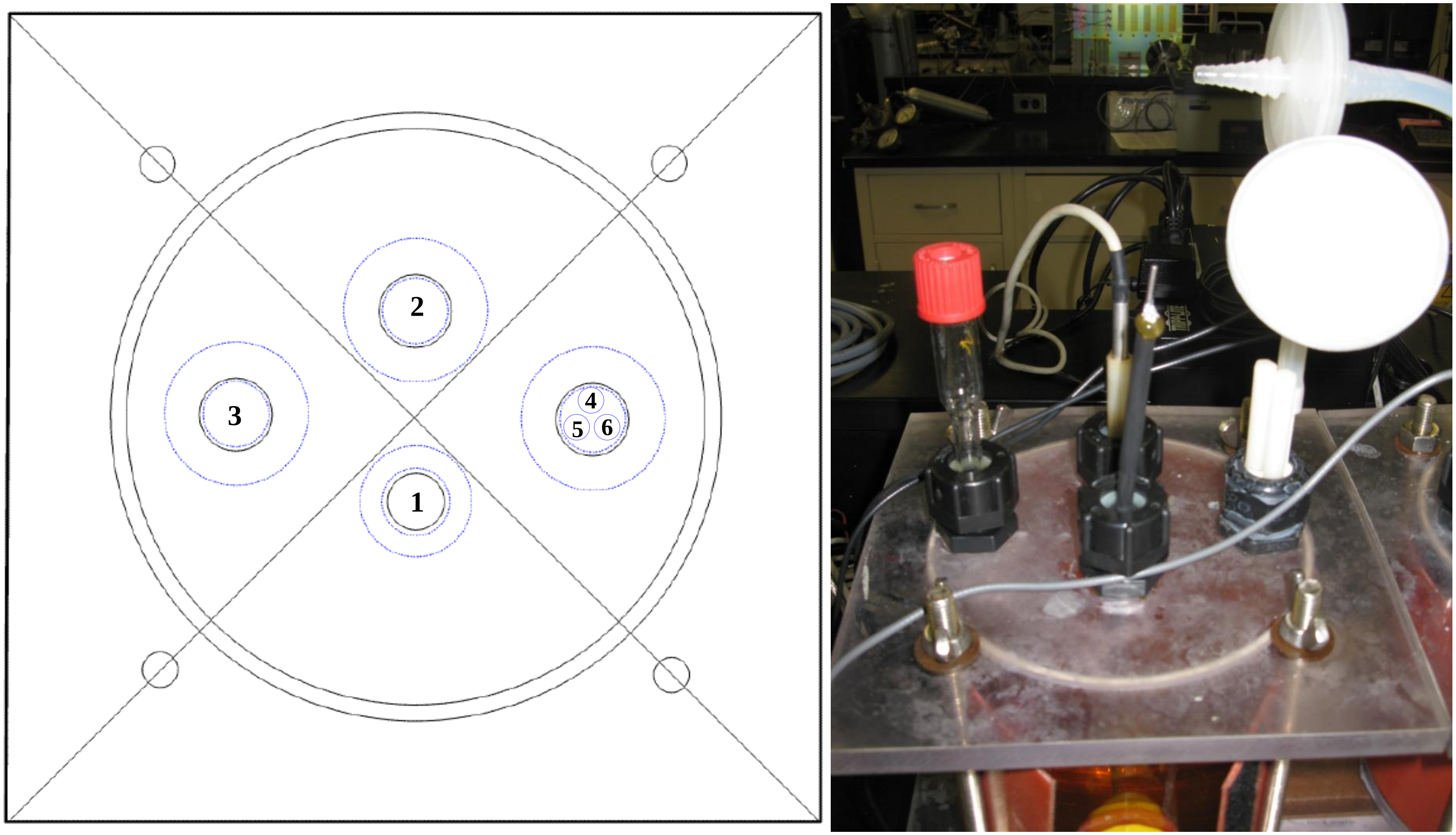


Figure 2. Two-Cell System for Testing CMM Membrane



Note:
1: Anode, 2: Micro pH probe, 3: Reference Electrode, 4: Medium Circulation Tube (In), 5: Medium Circulation Tube (Out), 6: Gas Tube (In)
7: Gas Tube (Out), 8: Graphite Rod, 9: Bulk pH probe, 10: Temperature probe

Figure 3. Anode Cell



Note:
1: Cathode, 2: Bulk pH probe, 3: Reference Electrode, 4: Gas Tube (In), 5: Gas Tube (Out), 6: Medium Tube (In)

Figure 4. Cathode Cell

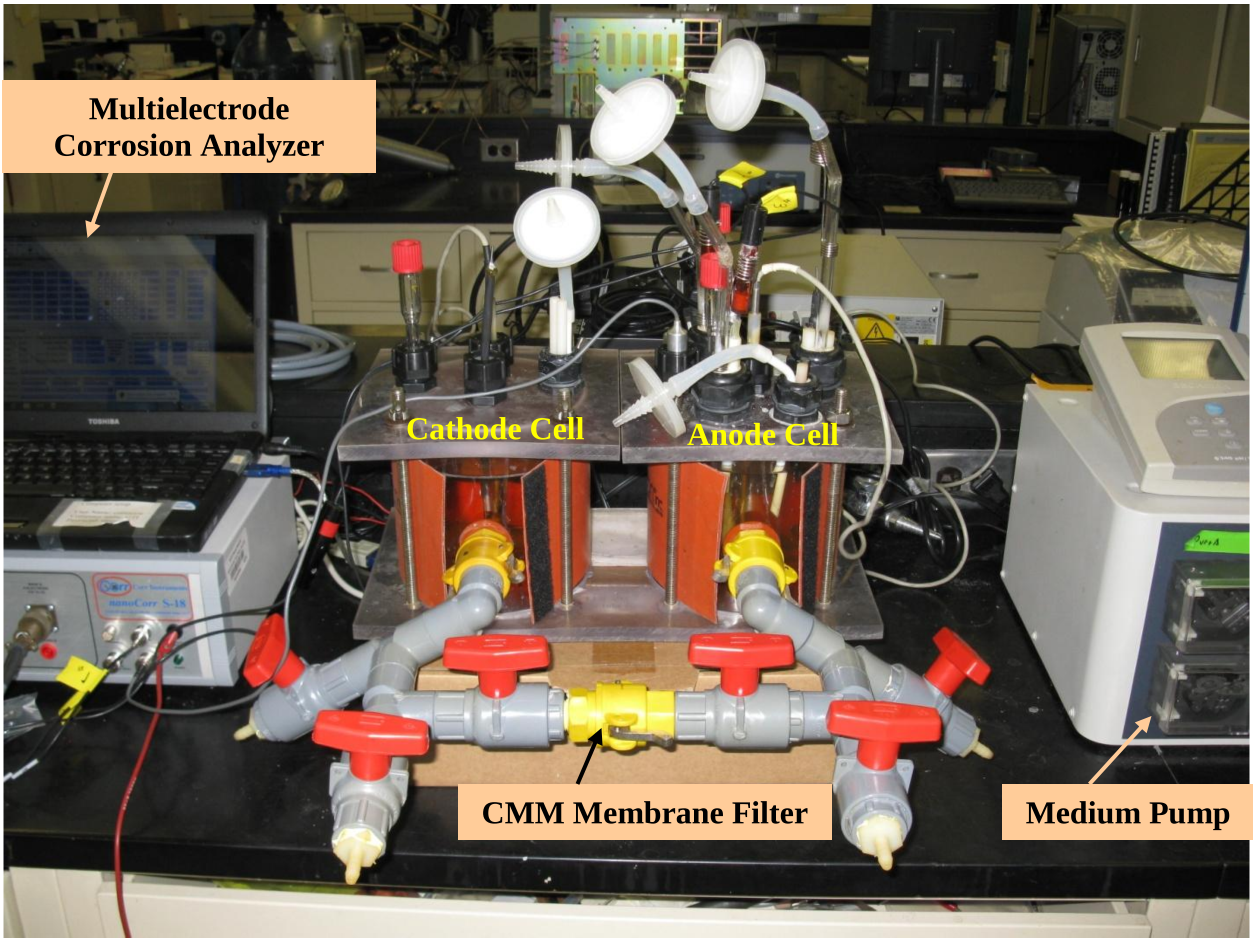
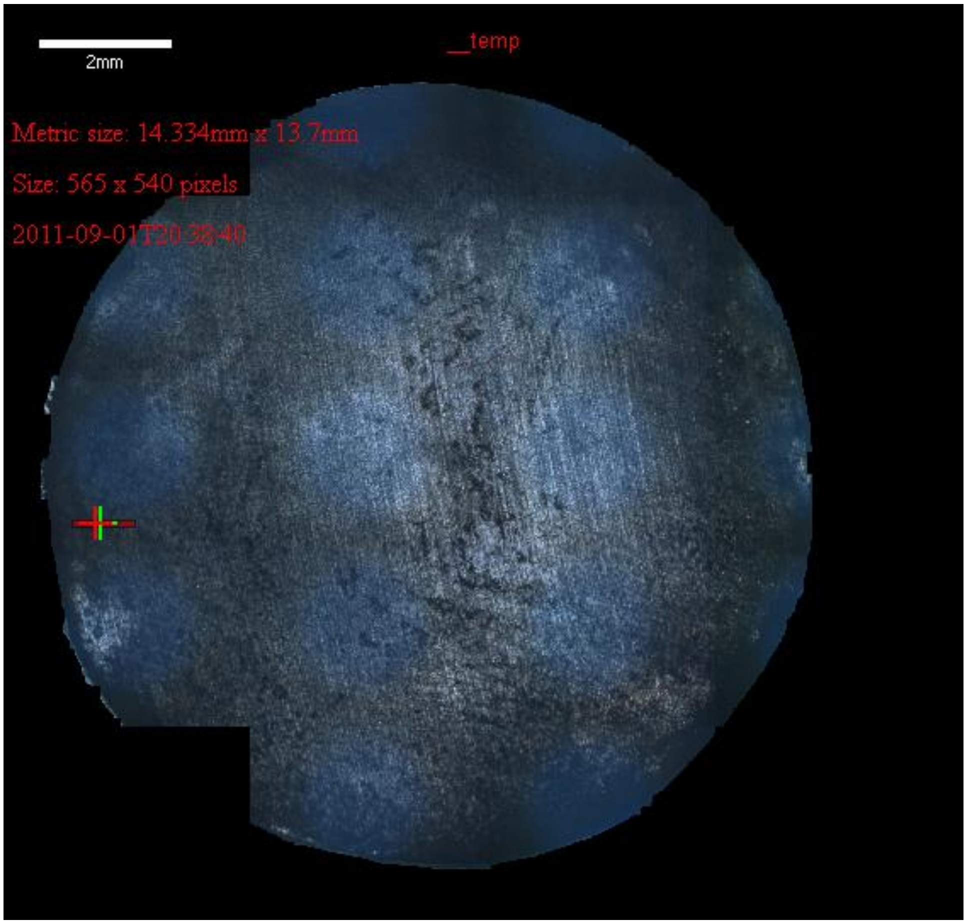


Figure 5. Dual Cell Electrochemical Test Setup



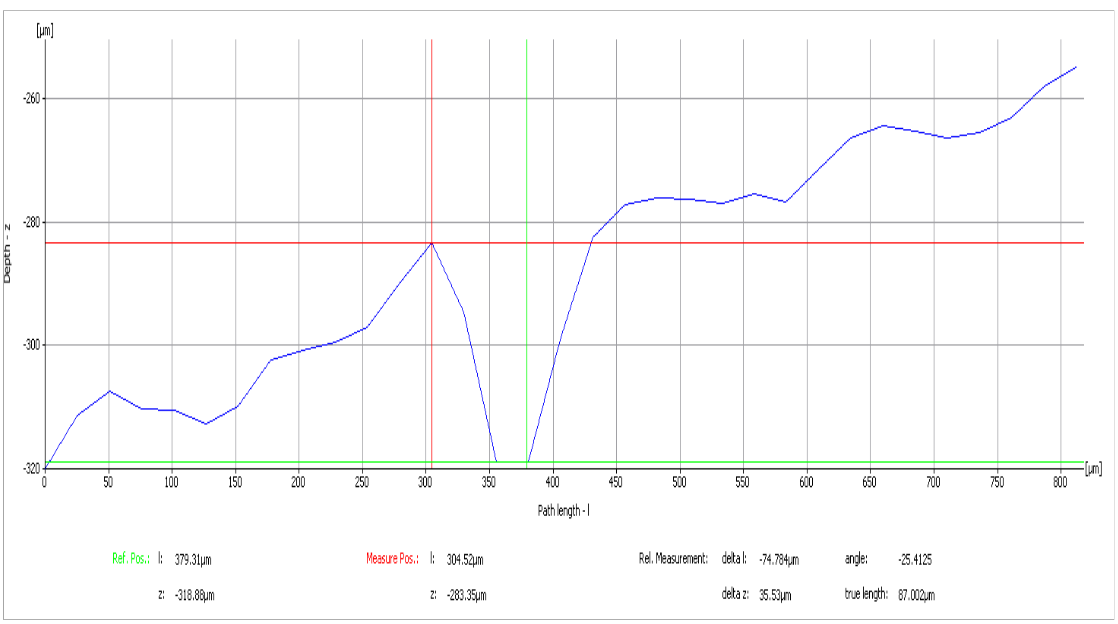


Figure 6. Pit Depth at 37^{o}C (The Largest Pit Depth Was 35µm).

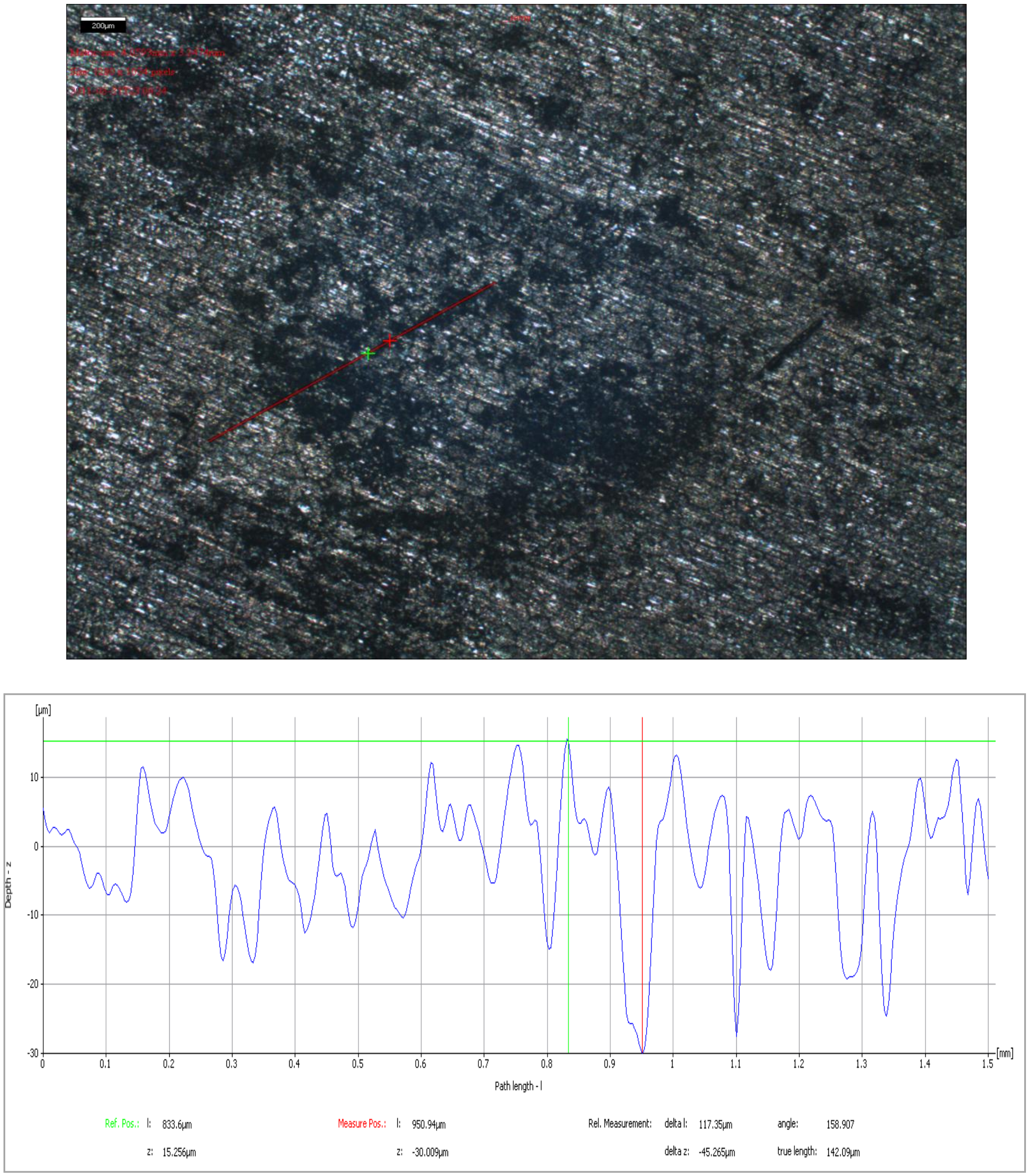


Figure 7. Pit Depth at 50^{o}C (The Largest Was 45µm)

Figure 8. Collecting Landfill Gas

Figure 9. Collecting Landfill Gas

Figure 10. Collecting Dairy Farm Gas

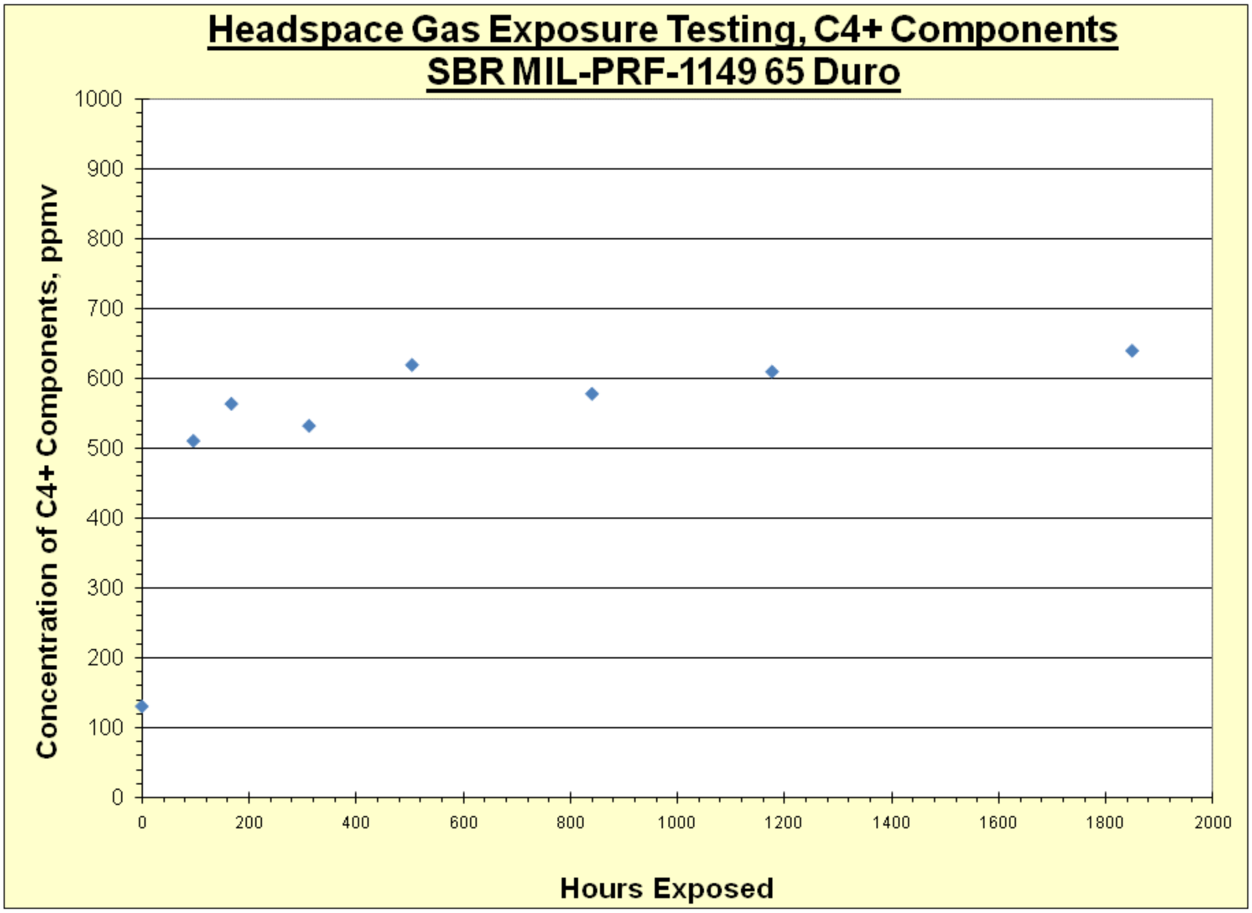


Figure 11. C4+ Head Space Test Results for SBR in Natural Gas Saturation Test

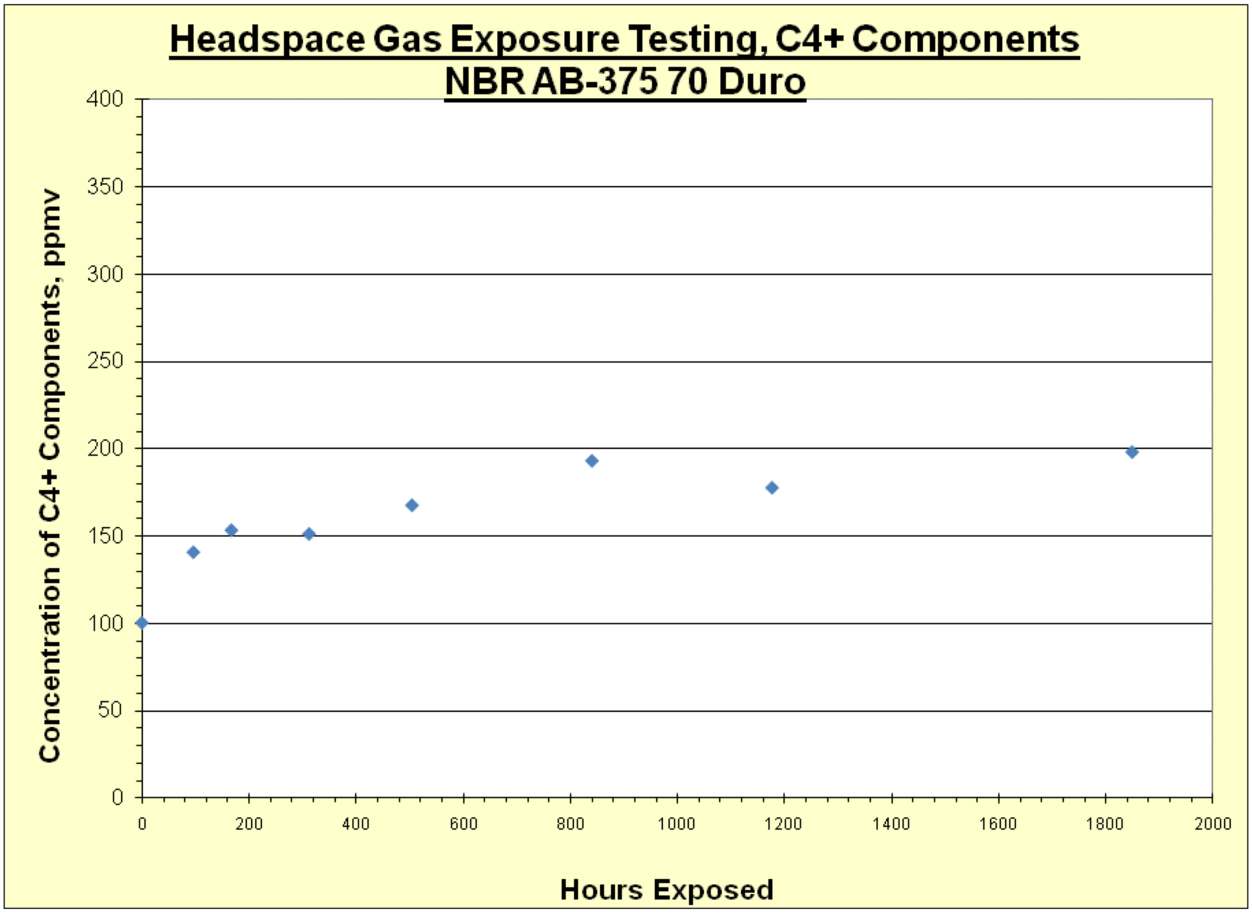


Figure 12. C4+ Head Space Test Results for NBR in Natural Gas Saturation Test

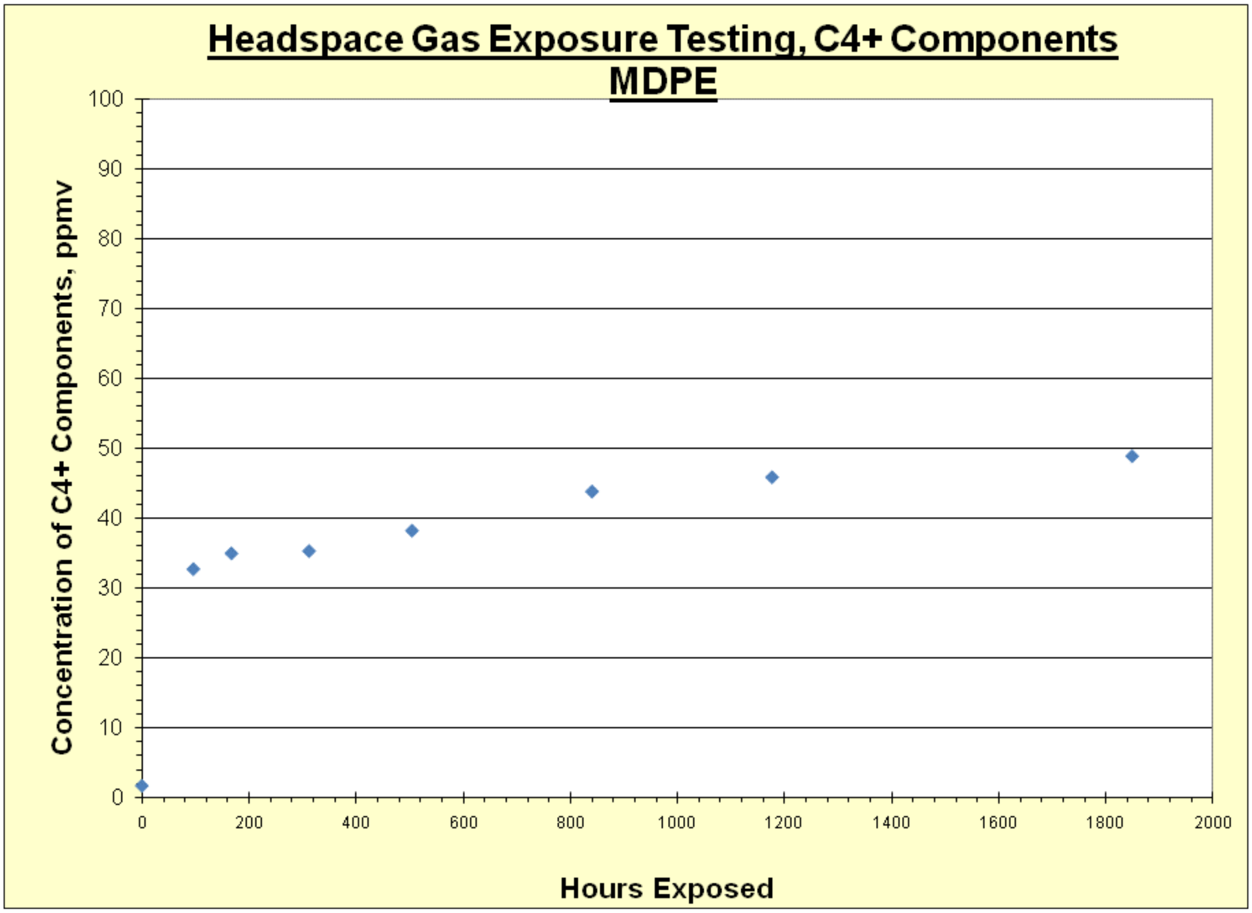


Figure 13. C4+ Head Space Test Results for MDPE in Natural Gas Saturation Test

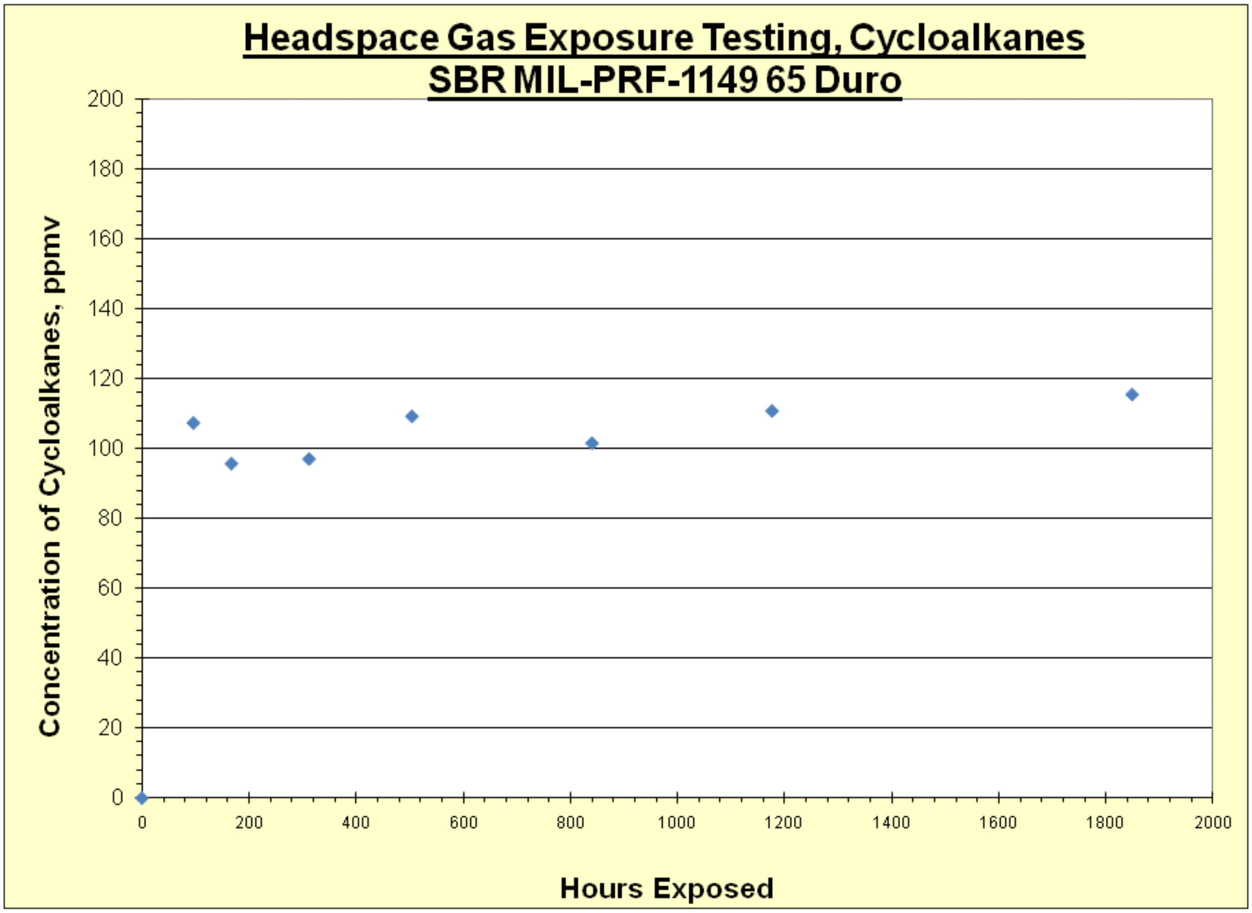


Figure 14. Cycloalkanes Head Space Test Results for SBR in Natural Gas Saturation Test

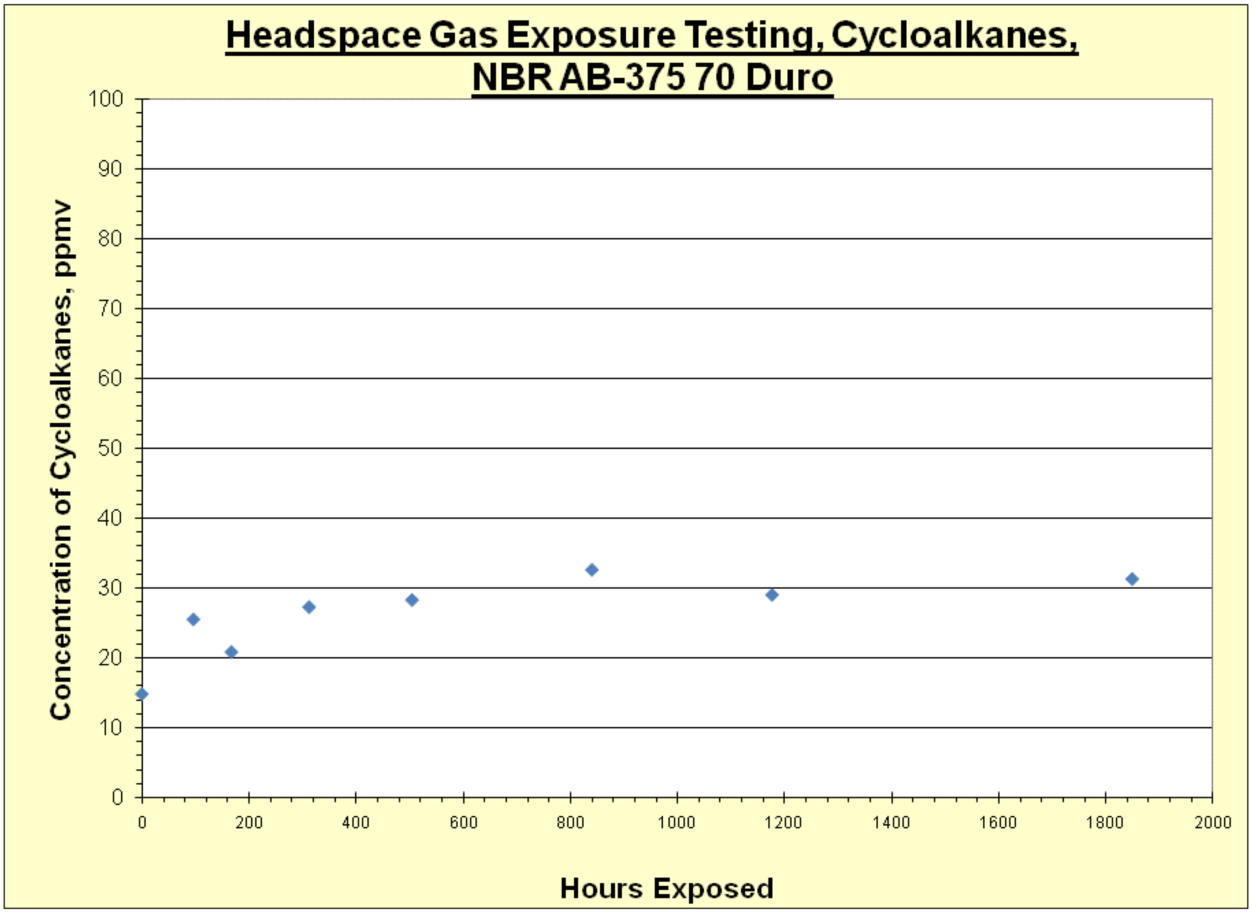


Figure 15. Cycloalkanes Head Space Test Results for NBR in Natural Gas Saturation Test

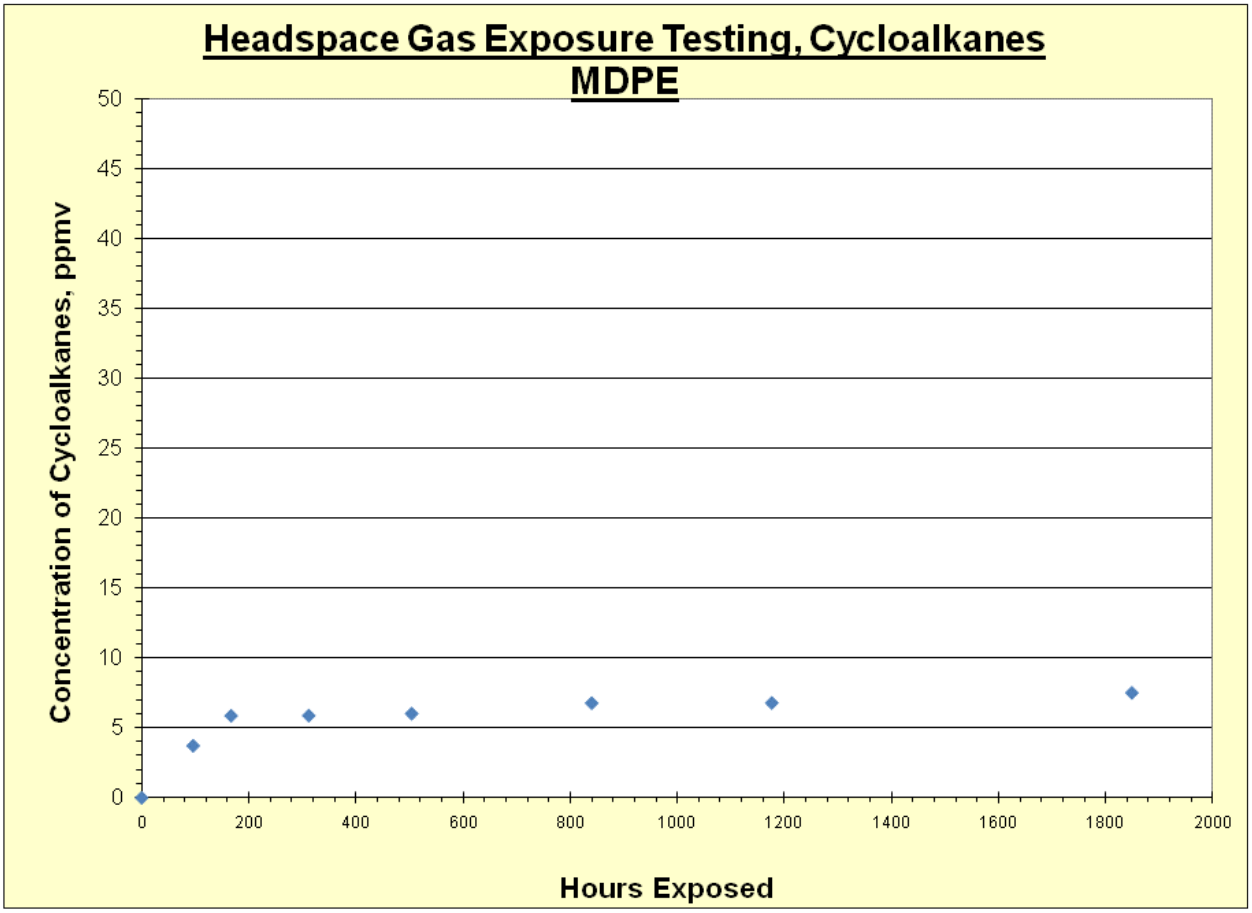


Figure 16. Cycloalkanes Head Space Test Results for MDPE in Natural Gas Saturation Test

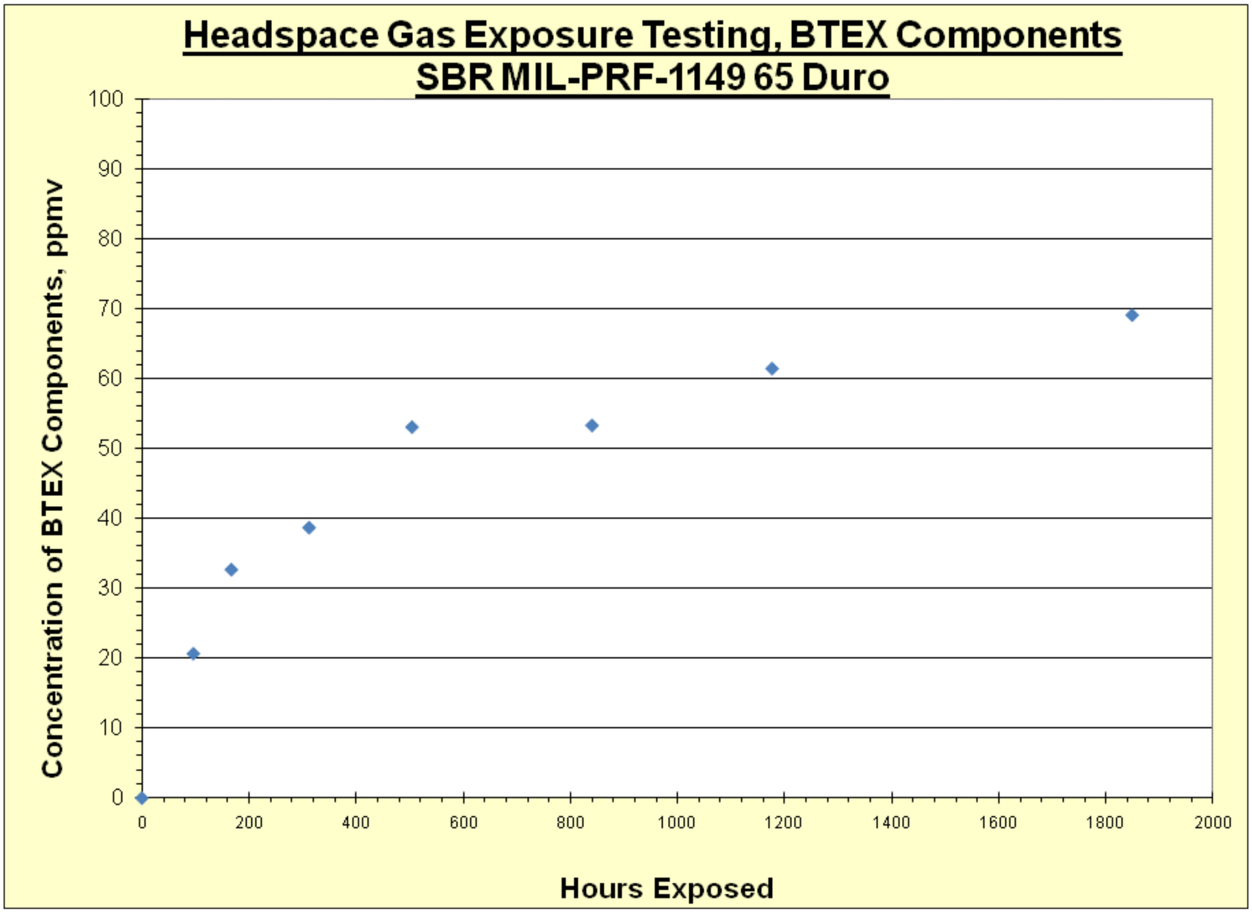


Figure 17. BTEX Head Space Test Results for SBR in Natural Gas Saturation Test

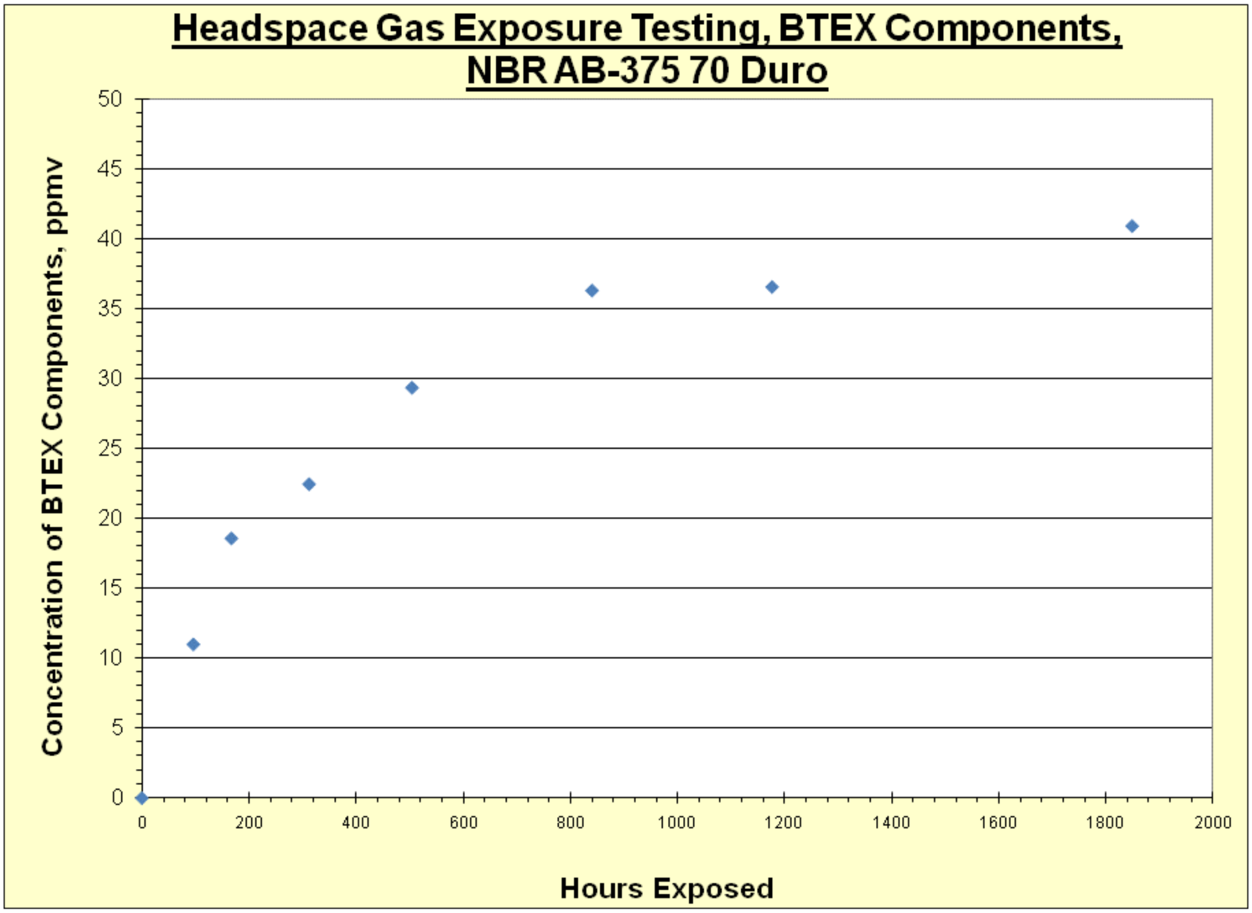


Figure 18. BTEX Head Space Test Results for NBR in Natural Gas Saturation Test

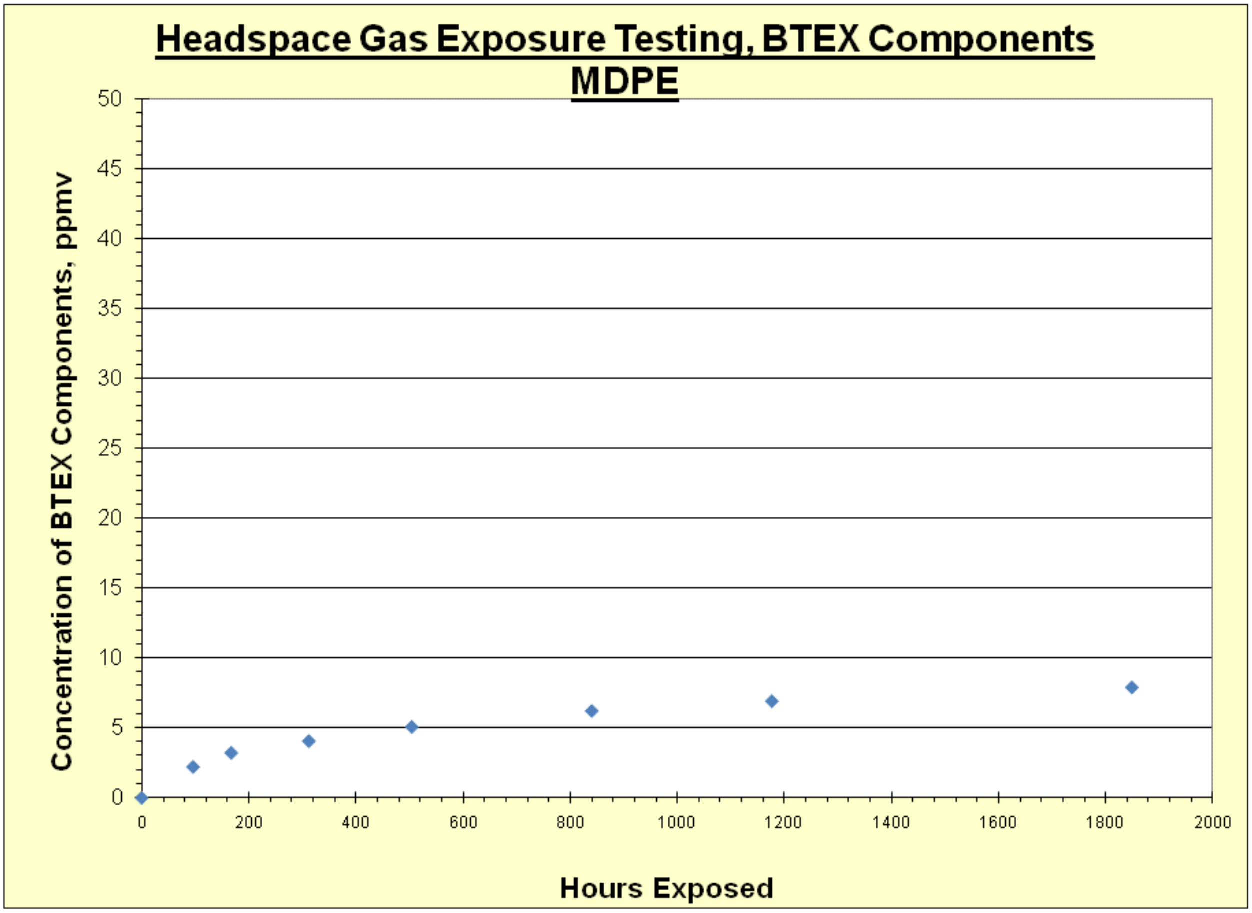


Figure 19. BTEX Head Space Test Results for MDPE in Natural Gas Saturation Test

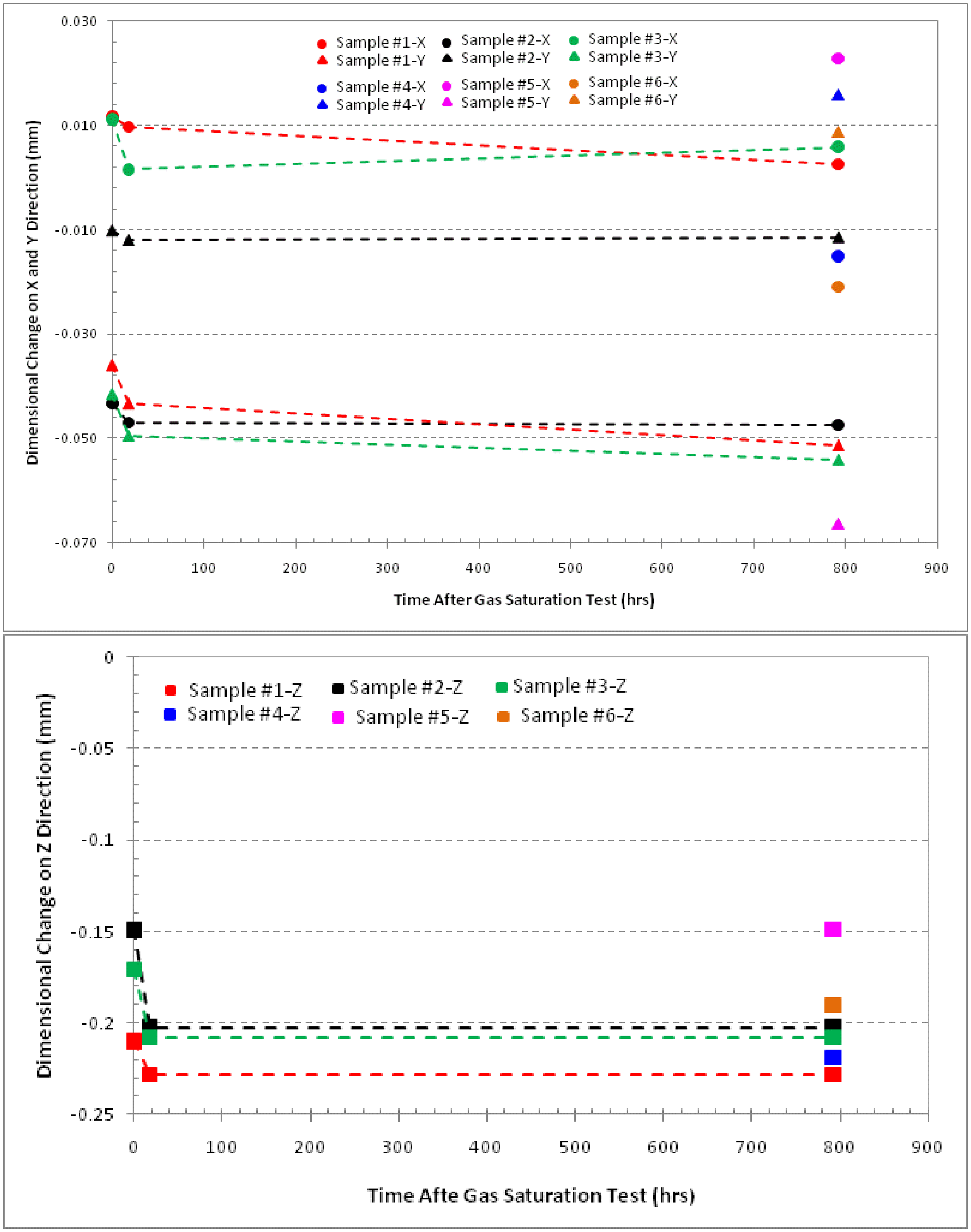


Figure 20. Dimensional Change of SBR Specimens on X, Y and Z Direction after Natural Gas Saturation Test (0, 18 and 792 hrs)

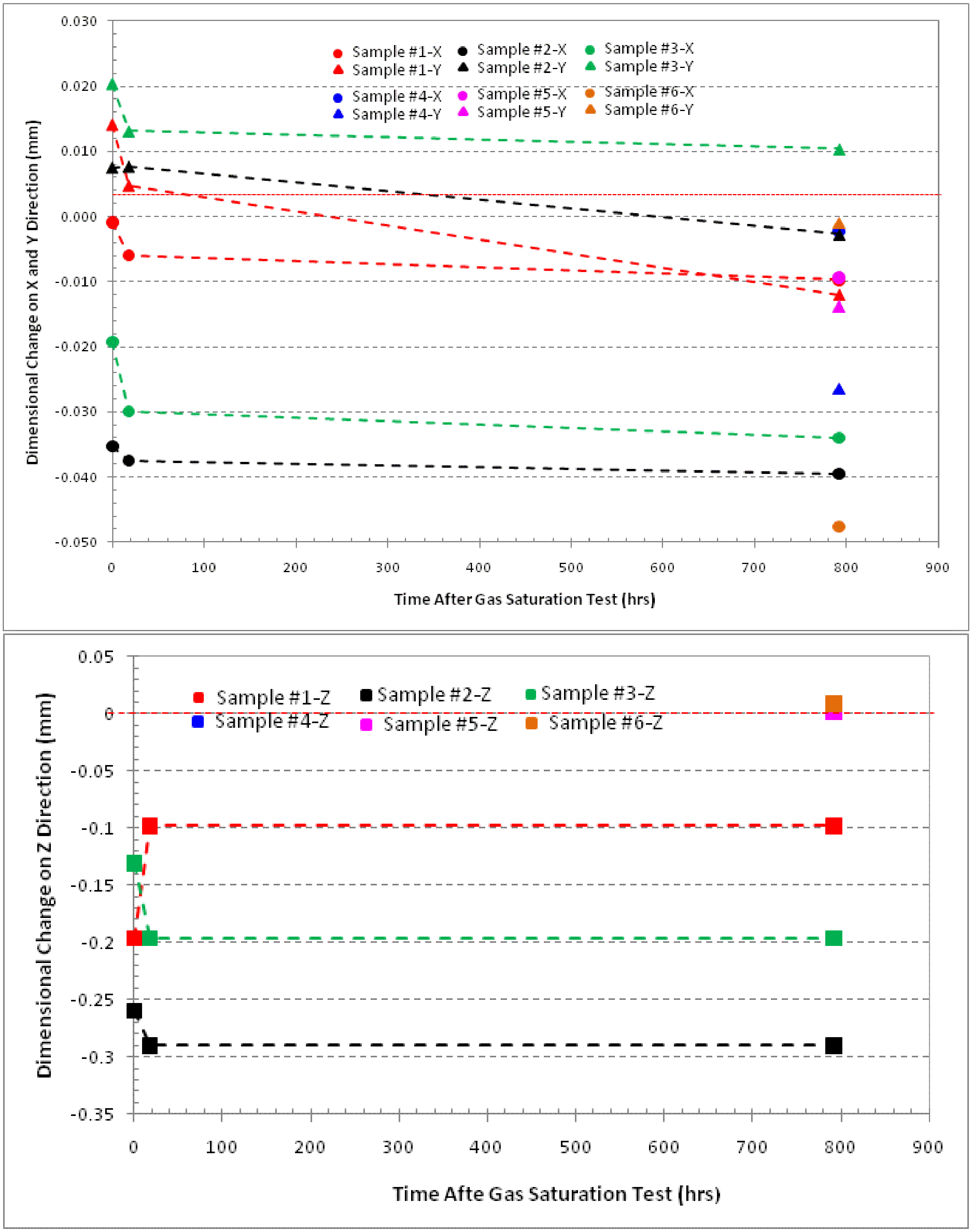


Figure 21. Dimensional Change of NBR Specimens on X, Y and Z Direction after Natural Gas Saturation Test (0, 18 and 792 hrs)

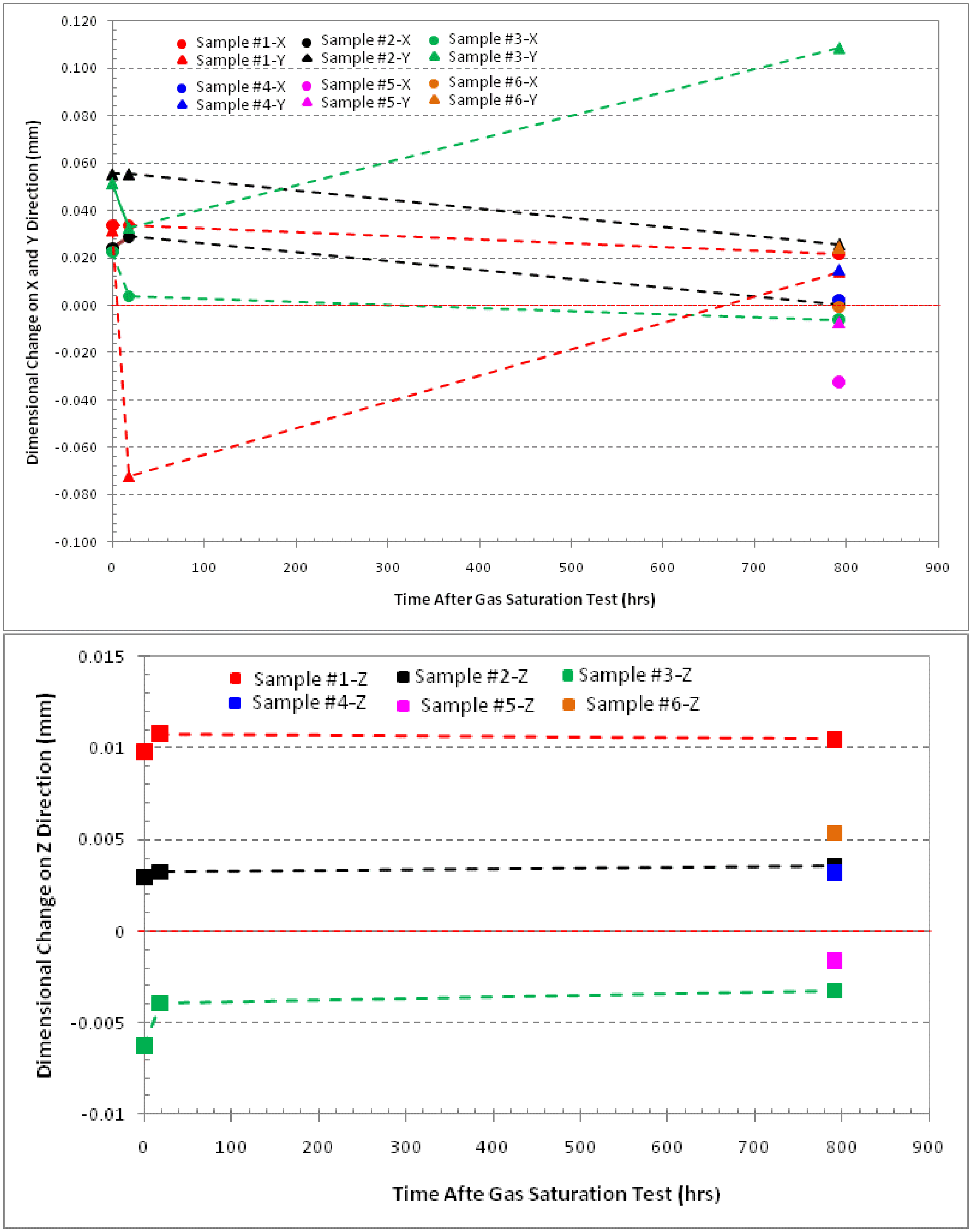


Figure 22. Dimensional Change of MDPE Specimens on X, Y and Z Direction after Natural Gas Saturation Test (0, 18 and 792 hrs)

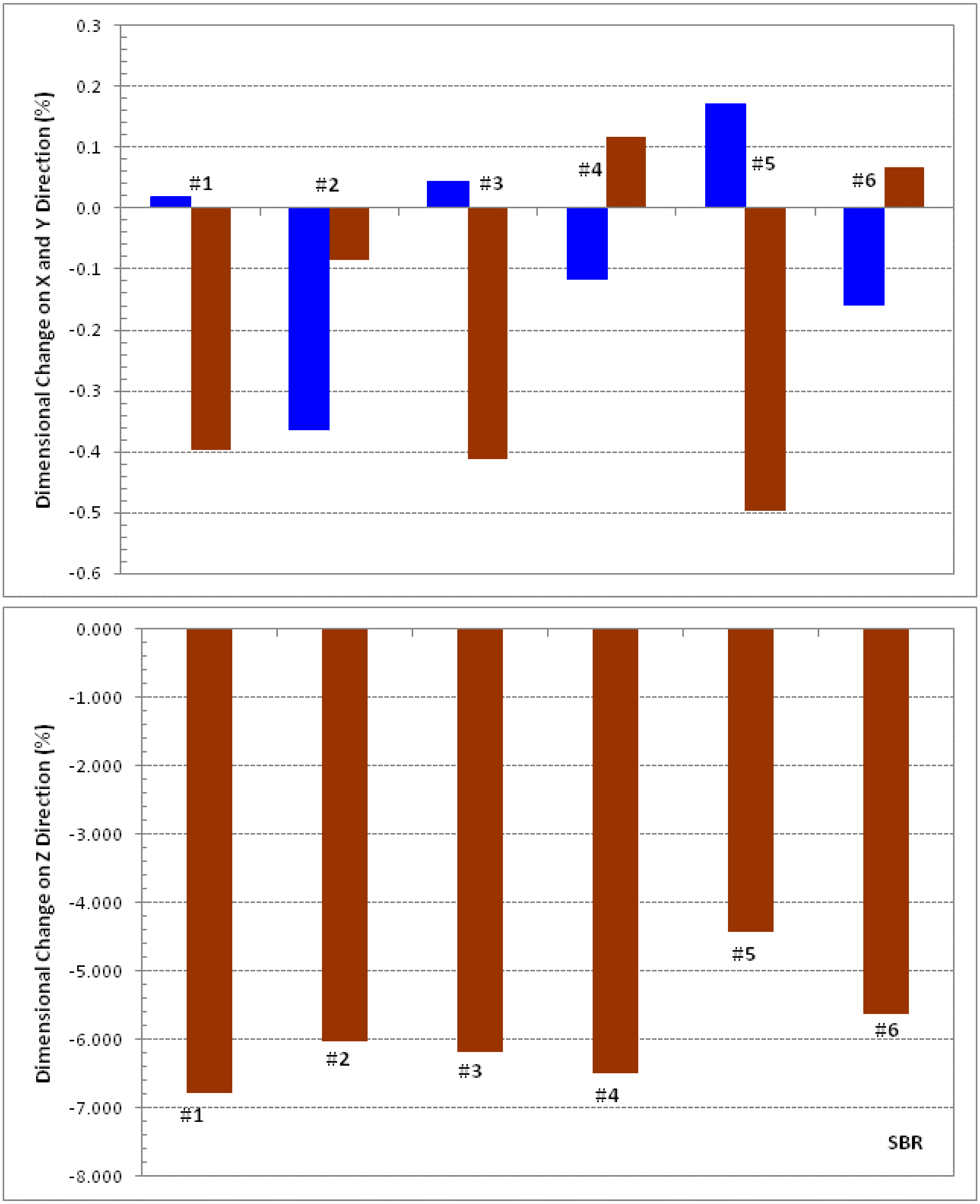


Figure 23. Dimensional Change of SBR (% of Baseline Dimension) Measured at 792 hrs after Natural Gas Saturation Test

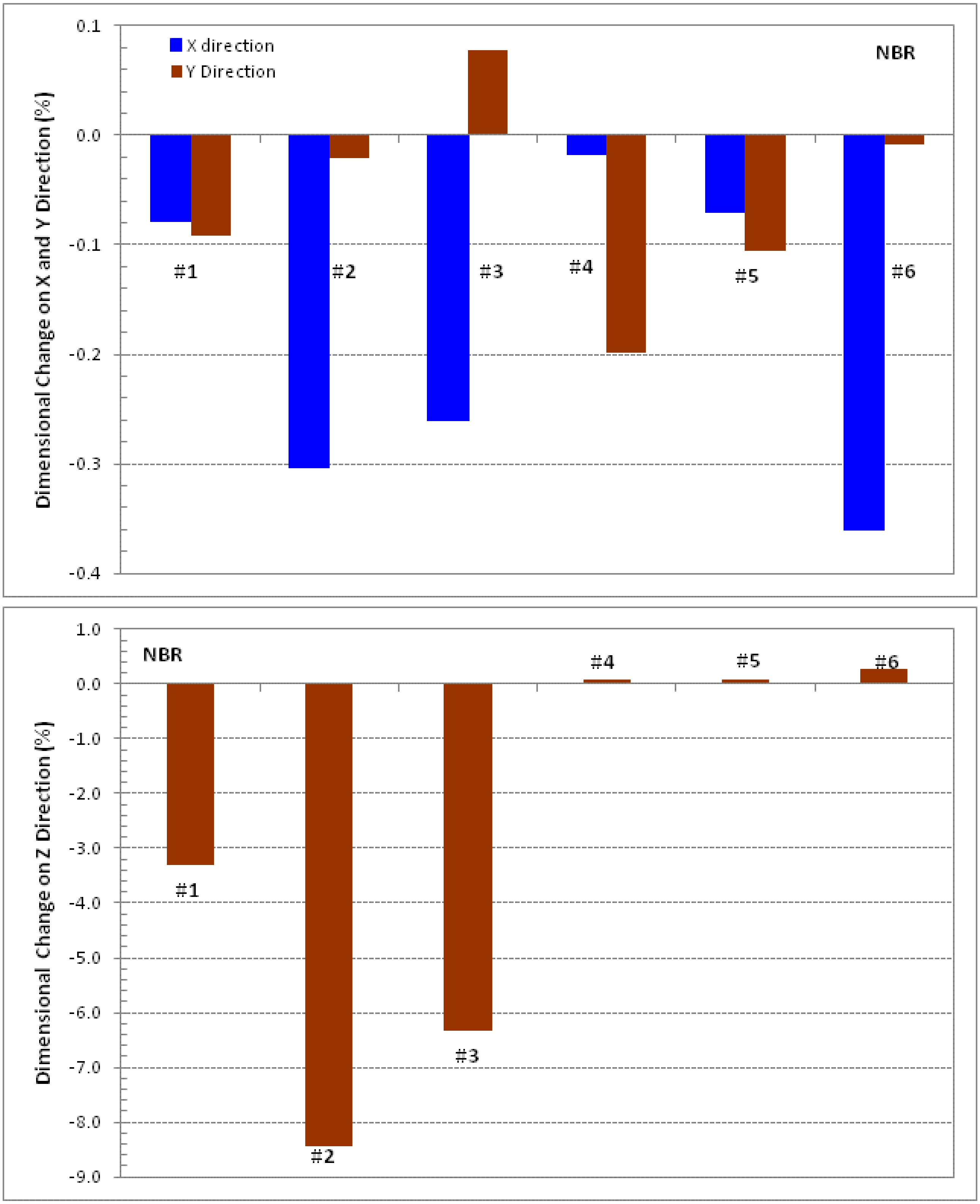


Figure 24. Dimensional Change of NBR (% of Baseline Dimension) Measured at 792 after Natural Gas Saturation Test

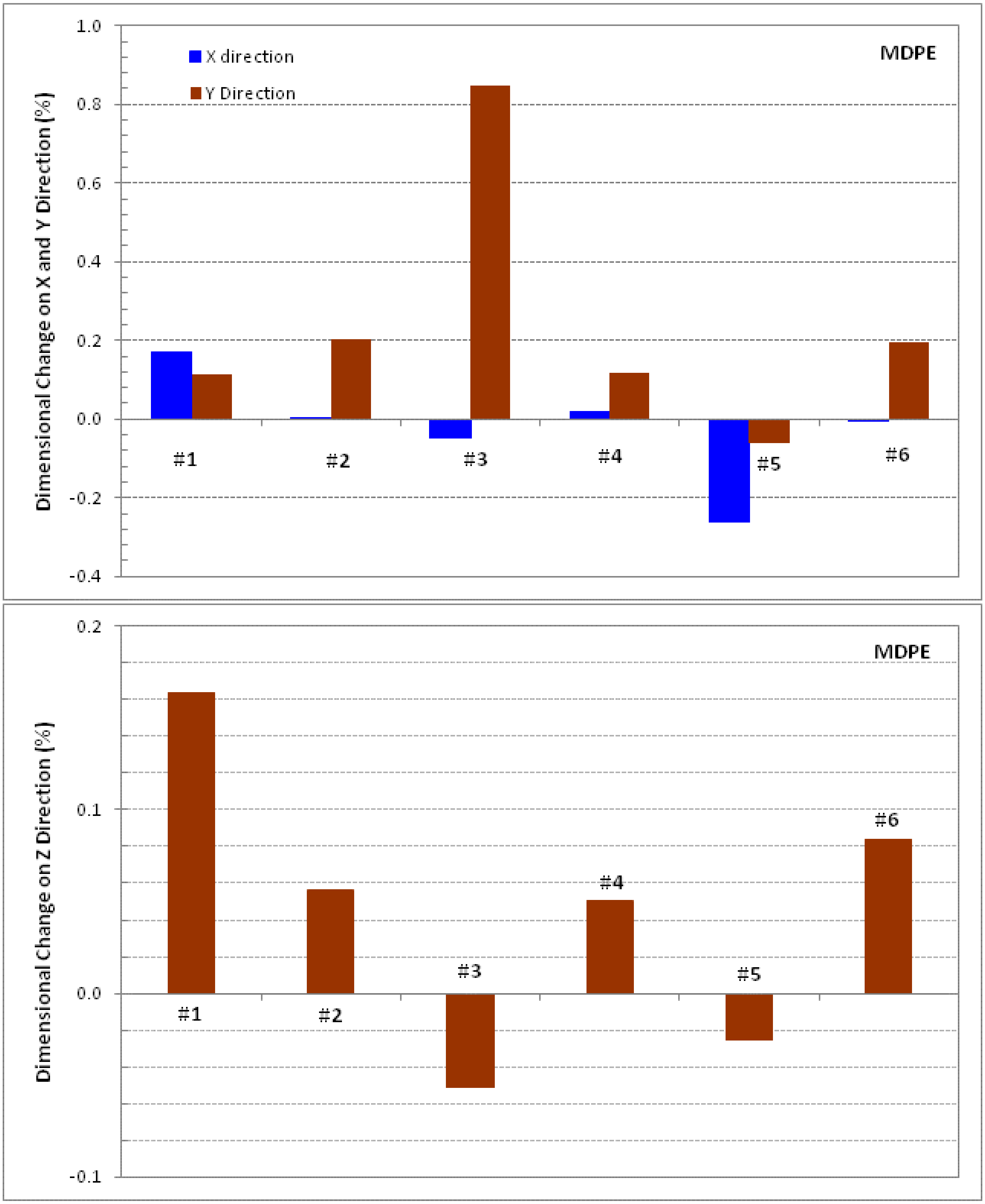


Figure 25. Dimensional Change of MDPE (% of Baseline Dimension) Measured at 792 after Natural Gas Saturation Test

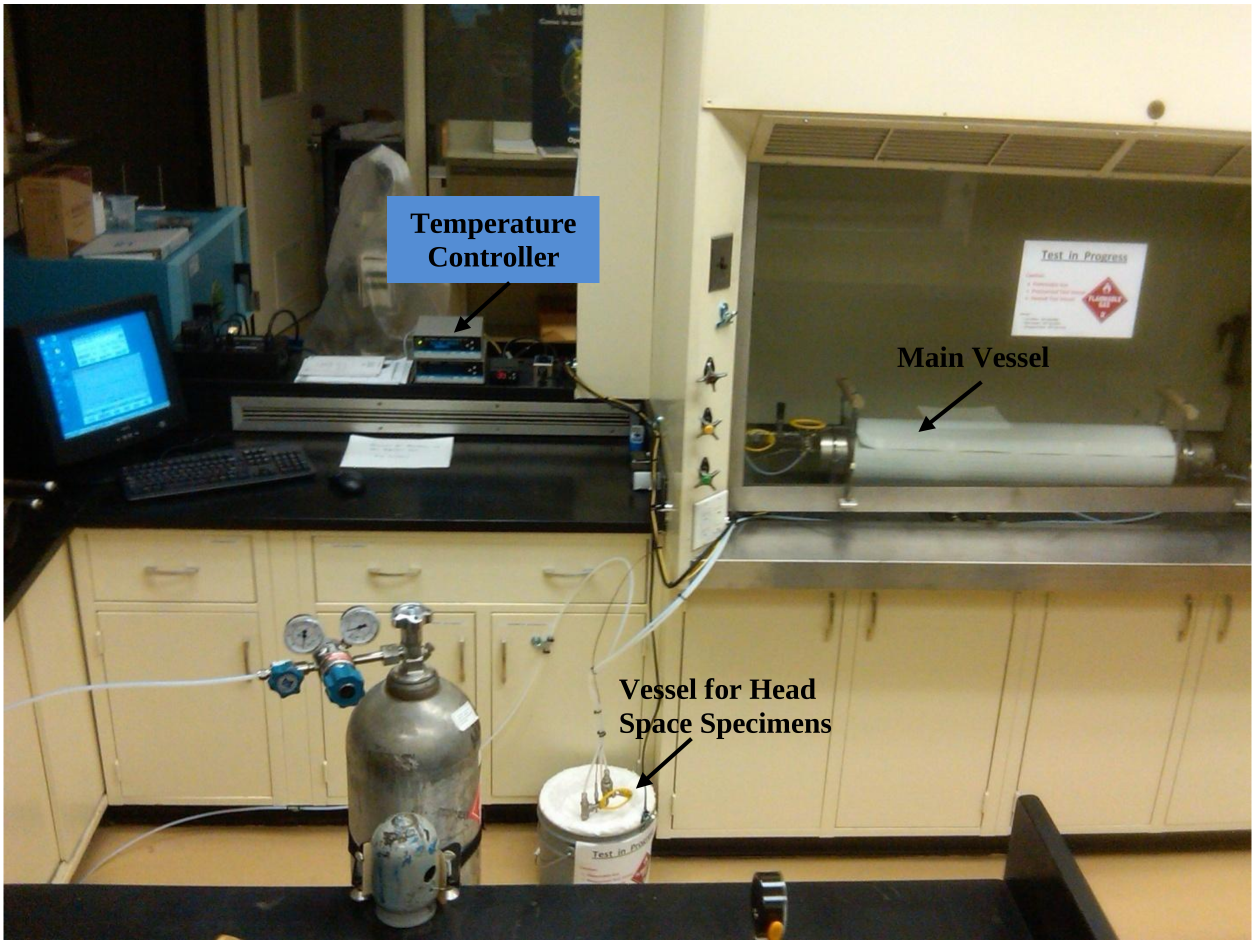


Figure 26. Gas Saturation Test System

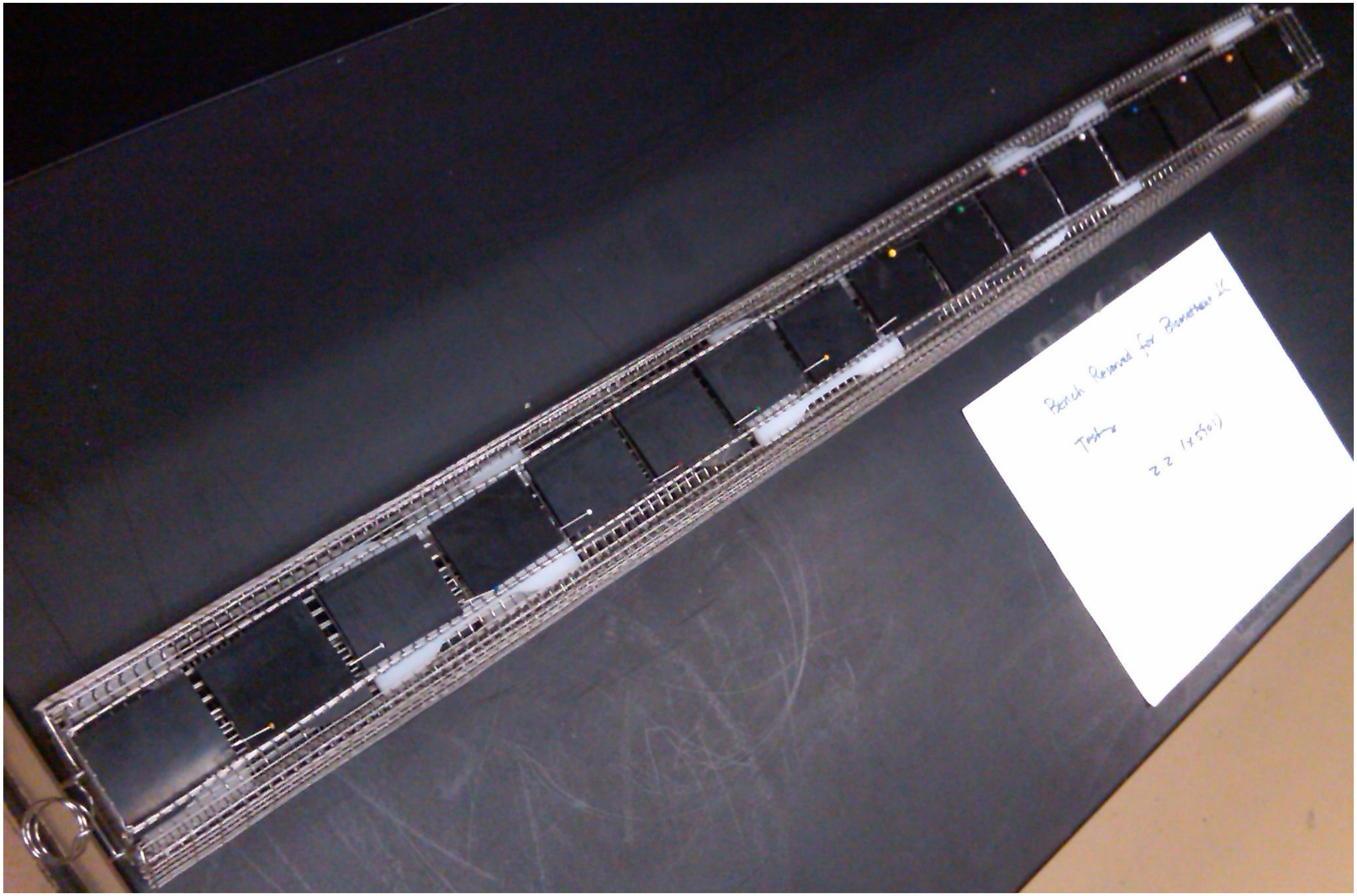


Figure 27. The Test Specimens in the Cage before Loading into the Pressure Vessel

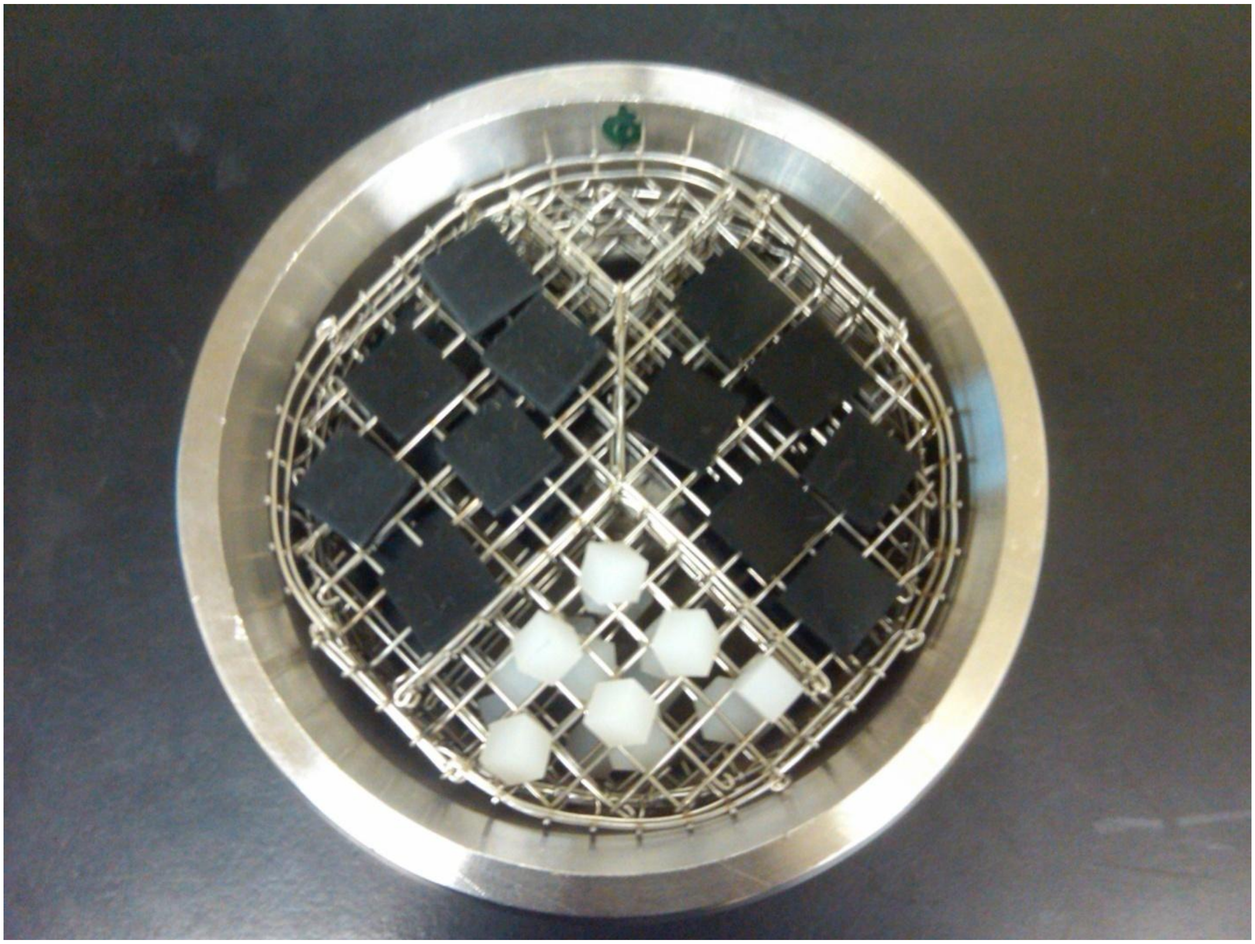


Figure 28. Head Space Test Specimens Loaded in the Vessel

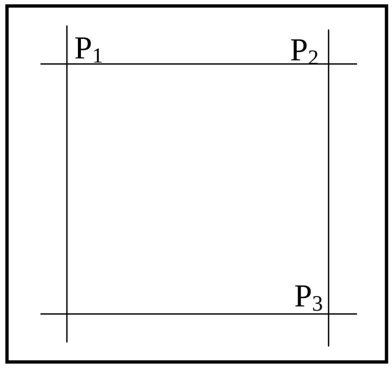


Figure 29. Dimensional Measurements on X (P_1P_2) and Y (P_2P_3) Directions